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Volume IV

PRODUCTION OF JET FUELS FROM COAL DERIVED LIQUIDS

VOL IV - GPGP JET FUELS PRODUCTION PROGRAM-FEED ANALYSES COMPILATION AND REVIEW

R.J. ROSSI

BURNS AND ROE SERVICES CORPORATION
SCIENCE APPLICATIONS INTERNATIONAL CORPORATION
PITTSBURGH, PA 15236

JULY 1988

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In September 1986, the Fuels Branch of the Aero Propulsion Laboratory at Wright-Patterson Air Force Base, Ohio, commenced an investigation of the potential of jet fuel production from the liquid by-product streams produced by the gasification of lignite at the Great Plains Gasification Plant (GPGP) in Beulah, North Dakota. Funding has been provided to the Department of Energy (DOE) Pittsburgh Energy Technology Center (PETC) to administer the experimental portion of this effort. This document reports the results of the effort by Burns and Roe Services Corporation/Science Applications International Corporation (BRSC/SAIC to compile and review physical and chemical characterization data for the GPGP by-product liquids. This report describes the relative reliability of the various characterization data and indicates where specific limitations exist. Finally, the report also serves as a general reference for comparing and assessing the results of future GPGP liquid by-product analysis.

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This report has been reviewed by the Office of Public Affairs (ASD/PA) and is releasable to the National Technical Information Service (NTIS). At NTIS, it will be available to the general public, including foreign nations.

This technical report has been reviewed and is approved for publication.

WILLIAM E. HARRISON III, Fuels Br

CHARLES L. DELANEY, Chief
Fuels Branch

FOR THE COMMANDER

BENITO P. BOTTERI, Assistant Chief
Fuels and Lubrication Division

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FOREWORD

In September 1986, the Fuels Branch of the Aero Propulsion Laboratory at Wright-Patterson Air Force Base, Ohio commenced an investigation of the potential of production of jet fuel from the liquid by-product streams produced by the gasification of lignite at the Great Plains Gasification Plant in Beulah, North Dakota. Funding was provided to the Department of Energy (DOE) Pittsburgh Energy Technology Center (PETC) to administer the experimental portion of this effort. This report details the effort of Burns and Roe Services Corporation/Science Applications International Corporation (BRSC/SAIC), who, as a contractor of DOE (DOE Contract No. DE-AC22-87PC79338), compiled and reviewed physical and chemical analyses conducted for these liquid by-product streams by other program participants. DOE/PETC was funded through Military Interdepartmental Purchase Request (MIPR) FY1455-86-N0657. Mr. William E. Harrison III was the Air Force Program Manager and Mr. Gary Stiegel was the DOE/PETC Program Manager.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF FIGURES, (CONTINUED)

LIST OF TABLES

LIST OF TABLES, (CONTINUED)

1. EXECUTIVE SUMMARY

The Great Plains Gasification Plant (GPGP) represents the first commercial Synfuels Plant in the United States. The plant is being operated for the Department of Energy by the ANG Coal Gasification Company to produce synthetic natural gas (SNG) from North Dakota lignite. Although the plant was designed to nominally produce 137.5 MMSCF of SNG per day, ANG has more recently operated at an average rate approaching 150 MMSCF of SNG per day. In addition to SNG, the GPGP also produces three liquid hydrocarbon by-product streams: Rectisol naphtha, crude phenol, and tar oil. Participants in the Jet Fuel from Coal Derived Liquids Program have analyzed samples of these by-product liquids periodically in order to assess the processing required to produce specification jet fuels and to assess the variability in product quality over time. Furthermore, ANG has sampled these liquids on a fairly regular basis since early 1985, primarily for marketing purposes. The purpose of this work is to collect all available physical and chemical characterization data performed for these liquid by-products during this program and assess the similarities and differences inherent in these data. Results of this effort are presented in the following report along with characterization data for the by-product liquids.

This study concludes that most of the basic physical and chemical analyses such as specific gravity, elemental composition, heating value, water content and ASTM distillation are generally reliable, although the ASTM D-2887 distillation method is preferred for the tar oil. Naphtha Reid Vapor Pressure (RVP), tar oil Conradson Carbon and GC component analyses for the naphtha, crude phenol, and tar oil also are reliable within certain limits. On the other hand, naphtha RVP can be particularly sensitive to changes in naphtha stabilizer operation or handling procedures due to its' high volatility.

Although GC analyses have verified the presence of guaiacol and catechol in the crude phenol, concentrations measured to date are

highly variable and require further definition. PONA (paraffin/olefin/naphthene/aromatic) analysis of the GPGP naphtha also varies substantially except for aromatics which comprise approximately 60 percent of the stream. While tar oil viscosity data at 120°F and 150°F appear to be reasonable, most crude phenol and tar oil data varies substantially and should be better defined. General observations of tar oil thermal instability by program participants emphasizes the need to collect more viscosity data, particularly regarding possible affects of time and temperature. Finally, exclusion of 1985 data has minimal effect on the statistical variation of the properties examined with exception of the naphtha which is much less volatile before July 1985 than afterwords. Overall, much useful data has been collected by participants in the Jet Fuels Production Program. Additional limited analyses such as GC/MS component characterization, acid/base extraction, proton- and C-13 NMR, and flash separation supplement these data and help direct additional analytical efforts, as well as, economic and process evaluations.

2. GOALS AND OBJECTIVES

As part of the Jet Fuels Production Program, the Department of Defense (DOD), and Department of Energy (DOE) have requested BRSC/SAIC to review and collect all available data that has been published and/or presented characterizing the liquid by-products produced at the Great Plains Gasification Plant (GPGP). As a product of this study, BRSC/SAIC has compiled these data to serve as a central reference to all participants in the Jet Fuels Production Program as well as other interested parties. During this program, Rectisol naphtha, crude phenol, and tar oil produced at the GPGP have been analyzed and characterized by participants to identify which streams or stream fractions are amenable to upgrading for jet fuel production.

In addition, efforts to enhance the profitability of the GPGP have been ongoing since plant startup. Consequently, ANG has been conducting a program to analyze the liquid by-products and to identify potential

marketing opportunities. These data have been made available for the Jet Fuels Production Program and, as a result, the overall GPGP liquid by-product data base is sufficiently large to provide a useful statistical basis for comparing analytical results from future efforts. An additional objective of this effort is to identify gaps and weaknesses in the existing data and thus provide guidance to future analytical work.

3. APPROACH

In order to develop the data base required for this study, BRSC/SAIC reviewed and compiled all available data pertaining to the analyses of the GPGP liquid by-products. Following this, a master list of all letters, reports, and handouts was prepared and reviewed with DOE and DOD to ensure that any pertinent documents were not overlooked. This list, includes analyses reported up to December 31, 1987 (References 1-40). Subsequently released data was not included in this report to expedite completion in a timely manner.

With this information at hand, a data base was assembled using Lotus 1-2-3 to collect the analyses most frequently conducted and reported. This includes ASTM distillation data, elemental analyses, density, HHV, and where available, vapor pressure, water content, and GC component analyses. BRSC/SAIC has performed statistical analyses where sufficient data are available to confidently identify and highlight significant similarities or differences. Other analyses, such as proton and C-13-NMR, and GC/MS are included where applicable. However, this report does not include detailed discussion of these analyses since the quantity of information available would result in an excessively long report. Consequently, these analyses are cross-referenced to facilitate retrieval of the original laboratory report. It should also be noted that it is not BRSC/SAIC's intent to interpret various analytical techniques.

Overall, the report itself is organized by by-product to permit separate discussion of the three by-product streams of interest: Rectisol naphtha, crude phenol, and tar oil. In order to place possible factors affecting product quality in context, a brief overview of the Lurgi Gasification Process is provided, followed by discussion of liquid by-product recovery facilities at the GPGP. Next, a general overview is provided of the types of analyses conducted on the three streams along with a brief discussion of the statistical technique used in this study. The report then provides a detailed evaluation of the by-product analyses, along with a summary of conclusions and recommendations from the evaluation.

4. LIQUID BY-PRODUCT PRODUCTION AT GPGP

The Great Plains Gasification Plant (GPGP) uses 14 Lurgi Mark IV Gasifiers to produce Synthetic Natural Gas (SNG) from Beulah-Zap lignite. Figure A-1 illustrates the proccessing sequence required to accomplish this. Likewise, Figure A-2 includes a simplified material balance for the base design case, which produces 137.5 MMSCFD of SNG. By-product rates for the base design are included for general reference. Moving bed (also known as fixed-bed) gasification processes such as that exemplified by Lurgi's dry-bottom Mark IV gasifier typically produce a wide-boiling liquid by-product which may or may not be recovered from the product synthesis gas. As illustrated in Figure A-3, the coal is charged to the top of the gasifier while oxygen and steam are fed countercurrently at the bottom of the vessel through a grate. Reaction of the oxygen with the coal in the combustion zone supplies heat driving the gasification reactions taking place in the zone immediately above the combustion zone. Heat is transferred between the "zones" by the countercurrent flow of hot gases produced by the reactions, while temperature is controlled by providing excess steam.[38,39] The steam also serves as a source of hydrogen (via water-gas shift and steam-carbon reactions) for the production of methane within the gasifier. As the gas stream flows upward through the coal, it heats and pyrolyzes the coal in the devolatilization zone. It is in

this area that the liquid by-products are formed along with additional light gases (CO_x, methane, ethane, H_2, H_2S, and NH_3). Since the temperatures in this zone are not high enough nor residence times long enough to decompose the hydrocarbonaceous materials, they exit with the gas stream into the drying zone. Finally, the remaining sensible heat contained by the combined combustion, gasification, and carbonization product dries the feed coal before exiting the gasifier.

The crude gas exits the gasifier at approximately 600°F and between 400-500 psig through a quench scrubber, where it is contacted with gas liquor to remove particulates. Following quench, the raw gas is sent to a waste gas exchanger as indicated in Figure A-4, and cooled to about 350°F to 370°F prior to further processing.[39] Gas liquor from the quench scrubber is sent to the gas liquor separation unit to recover tars and oils.

5. LIQUID BY-PRODUCT RECOVERY AND SEPARATION AT GPGP

Once the raw gas is quenched and cooled, this stream is split, with two-thirds going directly to the gas cooling block (Figure A-5). The remaining one-third is diverted to the Shift Conversion Unit where it is first shifted to produce a hydrogen-rich synthesis gas, then cooled to about 95°F before re-blending with the raw syngas. Cooling of the gas exiting the Shift Conversion Unit results in the condensation of tar- and oil-laden water streams which are sent to the Gas Liquor Separation Unit for further processing.

Cooling of the raw synthesis gas in the Gas Cooling Unit also forms condensates, commonly known as tarry gas liquor and oily gas liquor. These two streams are combined and transferred to the Gas Liquor Separation Area along with the Shift Conversion Unit condensates for further processing.[39] The cool raw gas is combined with the cooled gas from the Shift Conversion Unit and routed to the Rectisol Unit to remove H_2S and other sulfur compounds, CO_2 and hydrocarbons prior to final conversion to SNG.

5.1 Rectisol Naphtha

The Rectisol Unit utilizes methanol circulating at low temperature to contact the raw synthesis gas in an absorber. Naphtha is condensed in the cold methanol along with the acid gases and the small quantities of mercaptans and thiophenes present in the gas. As shown in Figure A-6, a number of sequential separation steps are required to recover lean methanol, naphtha, and the acid gases. Sulfur compounds and CO_2 are stripped from the condensed naphtha in the Naphtha Stripping Unit and sent to storage. The net result of the cleaning and separation steps taking place in the Rectisol Unit is a synthesis gas stream containing 0.2 ppm or less sulfur compounds. This gas stream, which now has the required 3.0 H_2:CO ratio and less than 2.0 ppm sulfur, is fed to the Methanation Area and is converted to SNG.

5.2 Crude Phenol

Gas liquor from the Gas Liquor Separation Unit contains considerable quantities of phenolic compounds which cannot be discharged without prior treatment. Since the concentration is too high to be degraded by conventional biological treatment, this stream is sent to a Phenosolvan Unit, where the phenols are extracted from the gas liquor with isopropyl ether (IPE) solvent. As seen in Figure A-7, this is accomplished in a series of mixer-settler tanks where a solvent-phenol mixture is produced by countercurrent extraction. This mixture is then sent to the Solvent Distillation Column and the Solvent Recovery Stripper to separate and recover the IPE, producing a crude phenol stream.

5.3 Tar Oil

As previously discussed, condensates from the cooling of the raw gas stream, otherwise known as gas liquor, are collected and sent to the Gas Liquor Separation Unit. These condensates contain tar,

tar oils, and dissolved compounds such as phenols, ammonia, carbon dioxide, and hydrogen sulfide. There they are further cooled, combined, and reduced in pressure. As illustrated by Figure A-8, the total stream is processed in a series of separators where the gas liquor separates into separate phases. Tar and tar oil are recovered from the gas liquor by gravity as the tar (sp. gr.>1.0), tar oil (sp. gr.<1.0), and water phase (sp. gr.≈1.0) separate into discrete phases.[5] Although it is not specifically a plant by-product, a dusty tar containing approximately 20 percent solids is recovered in the Primary Separator and recycled to extinction in the gasifiers.

A light, clean tar typically containing 2-6 weight percent solids is also recovered in the Primary Separators. Oil is recovered primarily in the Secondary Separators along with some tar carried over from the Primary Separators. The remaining aqueous phase, which still contains a small amount of oil is introduced into the Fuel Gas Liquor Separator, where the remaining oil is skimmed off and recovered. The oil-free water is then filtered and sent to the Phenosolvan Unit.

6. BY-PRODUCT ANALYSIS

While this report does not examine the accuracy and/or limitations of the analytical techniques applied by program participants, it is useful to briefly summarize the types of analyses performed on the naphtha, crude phenol, and tar oil stream to provide a general indication of the types of data that are available.

6.1 Rectisol Naphtha Analysis

For the Rectisol Naphtha stream, routine and seasonal analyses typically conducted by ANG includes the specific gravity, heating value, water content, and elemental composition. Additionally, the naphtha was frequently characterized using the ASTM D-86 dis-

tillation procedure. RVP, viscosity, PONA content, and component GC analyses are measured on a more limited basis. Amoco, WRI, and UNDEMRC have also conducted similar analyses on samples of the naphtha that they have received. UNDEMRC has also performed GC/MS analysis, carbon-13 (C-13) nuclear magnetic resonance (NMR) spectroscopy, and proton-NMR spectroscopy on the naphtha in order to provide a qualitative look at the types of compounds and functional groups present in this stream. Amoco[4] and UNDEMRC[23,28] have also performed limited analyses on "deodorized" or "sweet" naphtha samples from which the highly volatile, offensive-smelling mercaptans and thiophenes have been extracted. Lastly, WRI has also reported analyses of the naphtha stream, including specific gravity, elemental composition, GC/MS analyses and ASTM D-2887 GC distillation.[37]

6.2 Crude Phenol

Crude phenol analyses reported by ANG, Amoco, WRI, UNDEMRC, and HRI typically include measurements of properties such as specific gravity, water content, heating value, and elemental analyses. ASTM D-86 distillations were also frequently conducted and reported. Pour point, viscosity, and GC component analyses were also performed, although on a less frequent basis. WRI has performed D-2887 GC distillations on several crude phenol samples, while UNDERC and HRI have conducted true boiling point (TBP) distillations for this stream.[17,27,28] WRI has also reported the results of a flash distillation of the crude phenol conducted to prepare samples for tests examining the suitability of crude phenol fractions as feedstock for jet fuel production. Finally, HRI has performed GC component analyses while UNDERC has performed GC/MS, C-13 NMR, and proton-NMR analyses of whole crude phenol, and fractions from the TBP distillation of the crude phenol.

6.3 Tar Oil Analysis

As was the case with the Rectisol Naphtha and Crude Phenol streams, the GPGP Tar Oil analyses include measurements for properties such as specific gravity, heating value, water content, and elemental analyses. Some data are also reported for solids concentration in the tar oil. Since solids can plug catalyst beds, this data is important when designing upgrading facilities for this stream. Limited analyses regarding the specific composition of these solids are also available from Chromaspec, ANG, and WRI. Component analyses of the tar oil are also available, as are data on viscosity, vapor pressure, pour point, flash point, and Conradson Carbon. Because the tar oil contains heavy, high-boiling, complex hydrocarbons, distillation analyses was extended to utilize ASTM D-1160 vacuum distillation and D-2887 GC distillation techniques. The D-1160 and D-2887 methods provide more reliable information than the D-86, particularly regarding the last 30 percent of the tar oil, which tends to degrade in the D-86 test.[24,28] WRI also reported liquid-liquid extraction and elution chromatography results for the whole tar oil along with characterization data for a caustic-extracted tar oil they prepared for hydrotreating tests.[37] UNDEMRC has performed detailed analyses on the whole tar oil as well as acid, base, and neutral fractions of the tar oil. They have reported the results of GC analyses of these fractions along with short column separations determining the concentration of aliphatic, aromatic, and polar components. UNDEMRC has also reported the results of tests to quantify the stability of the GPGP by-products and in particular, the tar oil during D-86 distillation.[23]

7. STATISTICAL ANALYSIS OF GPGP BY-PRODUCT CHARACTERIZATIONS

Characterization data reported for the GPGP by-products have been compiled and subjected to statistical analysis to help determine the reliability of the data and its suitability for use in future evalua-

tions of product end uses (upgrading, extraction, etc.). Since distillation represents one of the most common analyses performed, significant emphasis focused on the evaluation of this data. However, other properties were evaluated and results reported where sufficient data existed to develop a reasonable statistical base. These data are summarized for the Rectisol naphtha, crude phenol, and tar oil in Appendix D, Tables D-1 through D-3, respectively.

The statistical analyses focus on the estimated 95 percent confidence interval for the data under consideration. This approach is frequently utilized when reporting experimental data in order to illustrate the relative variability and reliability of reported results. In general, a confidence interval can be determined from the following equation:[40]

$$\bar{Y} \pm t\, S_{\bar{Y}} = \bar{Y} \pm t \frac{S_Y}{n}$$

where:
- $\bar{Y}$ = sample mean for desired confidence level
- t = t distribution value for n-1 degrees of freedom
- n = sample size
- S_Y = sample standard deviation of Y data
- $S_{\bar{Y}}$ = estimated standard error of the mean

This type of analysis provides the user with a yardstick for assessing the reliability of the data they have collected. It can also serve as a basis for deciding whether or not to use a particular analysis in developing a process design or economic evaluation. For example, a recipient of a GPGP by-product sample can compare their analyses of the sample to that contained in this report to determine whether the material is representative. Similarly, this data can be used to assess the degree of flexibility required during design of specific design.

It is important to note that a portion of the data included in this report dates back to the 1985 operation of the Great Plains Gasification Plant. As operations continued, there have been changes in some operations as the staff became more familiar with the plant and as

economic conditions warranted. For example, ANG has demonstrated that the plant is capable of producing as much as 150-153 MMSCFD of SNG, as compared to the nameplate capacity of 137.5 MMSCFD. It is possible that some process operations have been modified to permit higher production rates and consequently, product rates and qualities may have changed as a result. At the suggestion of ANG,[14] BRSC/SAIC has evaluated the data with and without the 1985 analyses in order to assess whether changes in operation are reflected in the physical and chemical property data. It is also possible that the earlier analyses may reflect limitations in analytical techniques and/or apparatus that have been addressed as the participants have gained more experience handling and analyzing these streams. In general, it is probably desirable to give less emphasis to the 1985 data when specific questions arise regarding the representativeness of reported analyses.

Lastly, data reported by ANG for the period November 1985 through February 1986 represents no more than the repeat of the first analyses in the shipment log. Therefore, these reported data have been analyzed as only one data point, at the suggestion of ANG.

8. BY-PRODUCT DATA ANALYSIS RESULTS

8.1 Rectisol Naphtha

From a practical standpoint, Rectisol naphtha is probably the most difficult stream to accurately characterize of the three GPGP by-product streams. First and foremost, the naphtha contains a significant quantity of light, volatile materials which are easily lost. To make matters even worse, sufficient concentrations of mercaptans and thiols are contained in the volatile front end to result in noxious odor that makes physical handling of this stream extremely difficult. Nonetheless, it is potentially quite valuable, containing significant quantities of benzene, toluene, and xylene (BTX). Thus, it may have market value either as high-octane gasoline blendstock or raw material for petrochemical

production. Consequently, analyses of this stream is important, from the standpoint of evaluating end uses for this stream assessing possible deodorization techniques, and also to enable comparison between a researcher's sample and the actual typical naphtha produced at GPGP.

Review of the analyses performed by ANG, UNDERC, Amoco, and Chromaspec indicates that there is generally good agreement between the various analyses of the naphtha stream shown in Table 1. Average values as well as the 95 percent confidence limits for the analyses were calculated. Although the reported D-86 data cover a fairly wide range, further analyses of the Rectisol naphtha indicates the data agree fairly well, as evidenced by the relatively "tight" 95 percent C.L. shown in Figure 1 as well as Table 1. The only exception to this is the IBP through 10 vol percent distilled point which varies ± 10 percent and the final boiling point, which varies approximately ± 13 percent. Other properties such as specific gravity, heating value, water content and elemental analyses also show generally good agreement, although water and heteroatom content do vary more significantly. Given the volatility of the naphtha, the variation in nitrogen and sulfur content probably reflect losses of material, particularly the mercaptans and thiols to the atmosphere. This factor may likely be a cause of variation in the methanol, acetone, and methyl ethyl ketone content of the naphtha, where the 95 percent confidence level ranges between ± 19.7 percent to ± 37.4 percent.

PONA analyses vary to an even more significant extent due in part to the relatively low number of analyses conducted. Paraffin content varies by about ± 50 percent, while, olefins vary ± 112 percent, and naphthenes by about ± 54 percent. Surprisingly, aromatics concentration data exhibits relatively good agreement, averaging about 63.5 ± 3.4 LV percent. This represents a confidence interval which is ± 5.3 percent. Also, Amoco has noted the

presence of unstable diolefins in the naphtha which must be treated in order to meet product specifications. Individual component analyses indicate benzene, toluene, and xylene (BTX) concentrations equal 61.9 $\pm$5.0 wt. percent and thus comprise more than 90 percent of the aromatics in the naphtha. ANG has recently reported that they have measured BTX concentrations of 55 percent and 59 percent,[12] which, although less than the data quoted previously, are either within or very close to the 95 percent confidence limits. ANG has also commented that the heteroatoms contained in the naphtha stream can interfere with certain BTX analyses procedures and are willing to discuss their own experience with other laboratories.

Following discussions with ANG, it was also decided to analyze the data on a more rigorous basis. ANG has stated that the naphtha stabilizer was originally operated as a debutanizer,[16] thus removing light ends. As time has progressed, ANG has moved away from this operating mode, thereby leaving more low boiling components in the naphtha. Although overall analyses of D-86 distillation data agree fairly well, inspection of the D-86 distillation data reveals a marked difference in the first 30 vol percent of the naphtha for the samples collected between 03/01/85 and 07/19/85, as depicted in Figure 2. Statistical analyses of the modified data presented in Table 2 suggests the early data has relatively little impact on the overall analyses, except for the PONA analyses, where there are too few data points to attach any degree of confidence. The D-86 distillation data resulting from the exclusion of data collected through 07/19/85 generally falls into the 95 percent confidence interval calculated for the entire available data base, as shown in Figure 3. Further comparison of the reported data in Tables 1 and 2 highlights the narrowing of the data range that improves the D-86 data. A similar evaluation limiting the data to only that reported since 09/01/86 reveals a slight change in the D-86 distillation profile and other properties although most are still within the 95 percent C.L.

As previously mentioned, the raw Rectisol naphtha contains noticeable concentrations of sulfur compounds which result in extremely unpleasant odors, thus hindering or even preventing normal analyses. UNDEMRC and Amoco have both investigated procedures for deodorizing this stream with some degree of success. UNDEMRC has utilized two alternate methods: (1) sulfuric acid wash; and (2) caustic wash. The caustic wash was further refined by addition of copper sulfate and quinoline, which resulted in total deodorization. Amoco successfully used 20 percent caustic blended with a 50/50 methanol/water solution following limited results with caustic alone. Results of the Amoco NaOH and NaOH/MeOH extraction presented in Table C-1 directionally confirm the effectiveness of the extractions. Caustic alone removes about 27 percent of the sulfur, while the NaOH/MeOH combination removes 50 percent of the sulfur. The caustic seems to be somewhat more effective at removing nitrogen-and oxygen-containing compounds when used without the MeOH/water solution. Effects of the NaOH-only extraction are highlighted by comparison of the D-86 distillation in Figure 4. As can be seen, there is a significant difference in the first 30 percent of the naphtha, with the treated naphtha boiling 10°F-25°F higher than the raw naphtha.

UNDEMRC utilized both a sulfuric acid wash and a NaOH extraction to deodorize the Rectisol naphtha. Variations on both have been widely used in the refining industry to remove sulfur compounds and improve product quality. UNDEMRC has conducted GC and GC/MS analyses of untreated naphtha to identify the compounds present.[23] This data, presented in Table C-2, indicates that although the thiols, thiobismethane, and thiophene are not present in large quantities, there are more than sufficient quantities of these compounds to impart a noticeable and pronounced odor to this stream. Subsequent comparisons of GC/MS data for raw- and H_2SO_4-washed naphtha in Figure B-1 reveal that most of the thiophene has been removed. Results of the NaOH/$CuSO_4$ wash also achieved good results, as shown by the comparison of Figures B-2 and B-3. As

can be seen, most of the low boiling sulfur compounds and thiophenes have been removed, resulting is a significant reduction of odor. Addition of quinoline solution to the NaOH/$CuSO_4$ wash resulted in total elimination of the odor. It has been observed that the deodorization process also removes some of the C_2 and C_3 benzenes. While it is believed that the loss of these compounds is small and does not result in an economic debit, it is likely that the treating step would be optimized prior to design in order to avoid undesirable compound losses. UNDEMRC proton-NMR and carbon-13 NMR analyses presented in Table C-3 verify the highly aromatic nature of the naphtha.

Amoco and UNDEMRC have also attempted to quantify the volatility of the raw naphtha since it has been observed that there is a possibility of losing some compounds by vaporization, thus complicating analyses. In addition, vapor pressure is an important specification that must be met when blending naphtha to produce motor gasoline. It also is important information needed to design storage and handling facilities. Consistent with ANG's comment that the naphtha stabilizer was originally operated as a debutanizer, the RVP of naphtha produced in more recent operation are about twice as high as those measured early in GPGP operation (early to mid-1985). Furthermore, the RVP for naphtha subjected to caustic extraction by Amoco is only 5.5 psi, compared to the typical range of 8-11 psi measured by ANG, reflecting removal or loss of volatile light ends. Amoco also conducted tests using a "bomb" designed to retain light ends. Comparison of the C_5-contents of the sample collected using the "bomb" vs. a standard 1-gallon can are presented in Table C-4. Use of the can rather than the bomb results in the loss of all the C_3-'s, 60 percent of the C_4's and 12-50 percent of the C_5's.[2] This finding is further reinforced by WRI's observation that their sample of raw Rectisol naphtha contained 90+ percent BTX (Table C-5) as compared to the typical 55-65 percent level reported by other laboratories, suggesting that at least 30-40 percent of the WRI sample had been

lost. Comparison of Total Ion Chromatograms (TIC's) performed by WRI (Figure B-4) and UNDEMRC (Figure B-3) highlight the loss of light materials from the WRI sample.

Lastly, elemental analyses and D-2887 distillation data from WRI characterization of the Rectisol naphtha are presented in Table C-5. Since it appears that the sample was not representative of the "typical" raw naphtha due to the loss of light boiling compounds from the front end, the data were not factored into the statistical analyses. However, the D-2887 data have been presented in graphical form (Figure 5) for reference.

8.2 Crude Phenol

As was the case with the Rectisol naphtha, GPGP crude phenol has been analyzed by ANG, Amoco, UNDEMRC, WRI, and Chromaspec. In addition, HRI has also analyzed the crude phenol as part of their test program to assess the ability of the Dynaphen process to produce pure benzene and phenol. Statistical analyses of the characterization data, presented in Table 4, indicate that most measurements agree reasonably well. Major exceptions are items such as the viscosity data, where large variation in a relatively small sample population result in a 95 percent confidence interval that is essentially meaningless. Similar variations are exhibited by some of the component analyses, such as BTX/Light Aromatics, methoxyphenol and the diol isomers. This may reflect the difficulty associated with conducting GC analysis on a stream which contains many highly polar oxygenated compounds. On the other hand, the fairly large variations in API gravity and sulfur content (95 percent confidence interval = $\pm$ 31 percent) are likely due to taking differences between small numbers. In addition, sulfur and oxygen analyses are sometimes subject to an inherent variability due to analytical technique. However, the D-86 distillation data exhibit very good agreement as shown in Figure 7, where the 95 percent confidence interval is within $\pm$5 percent,

except for the front end. The somewhat greater variability of the IBP and 5 percent distilled points are not surprising given the sharpness of the increase in boiling point in that region.

Statistically speaking, analyses of crude phenol samples taken during 1986 and 1987 are very similar to the data based on the entire 1985-87 sample. As seen in Figure 8, the D-86 distillation results are basically no different than that from the 1985-87 data. The other characterizations are also quite similar as illustrated in Table 5. The only surprise is the viscosity data collected at 210°F, which has a 95 percent C.L. that reflects relatively little variation. The 1986-87 sample has an average viscosity which is 68.8 ± 14.5 cst whereas the 1985/87 sample was 148.9 ± 238.4 cst. In fact, the approximate 95 percent confidence intervals for the 1985/87 sample were so large as to be meaningless.

HRI and UNDEMRC have performed TBP distillations on the crude phenol which are in generally good agreement although there are some differences. As illustrated in Figure B-5, the HRI crude phenol contains about 15 wt percent 350°F$^-$ material while the UNDEMRC sample apparently contains approximately 8 wt percent 350°F$^-$ material. The only other difference is the indication that the UNDEMRC sample gradually ramps up from 360°F to 380°F-385°F whereas the HRI sample exhibits more of a step change at the 52-55 percent distilled region. There does not appear to be any particular explanation for the differences other than general sample variability, possible changes in GPGP Phenosolvan unit operation, and potential differences in the TBP analytical procedures.

WRI has also reported results of GC distillations (ASTM D-2887) performed on crude phenol samples. The results are reported in Figure B-6. Since the GC distillation approximates a true boiling point distillation provided representative standards are used, a

GC distillation can be used in place of a TBP for most comparisons. However, the GC distillation data are presented on a volume basis, while the TBP data are on a weight basis. Consequently, specific gravity for each fraction is required to put the data on a consistent, comparable basis. Since this data is not readily available, the TBP and GC distillation data have not been compared.

On the other hand, D-86 and D-1160 data can be converted to TBP and vice-versa using standard API procedures. This was done for comparison between the average D-86 data, the May 1985 Chromaspec D-1160 analysis, and the D-2887 GC distillation data from WRI. These comparisons, presented in Table D-2, Appendix D, agree fairly well but are limited in value due to the lack of definition of the front-and back-end tails, as well as the small sample population.

UNDEMRC, WRI, HRI, and ANG have performed additional analyses on the crude phenol such as proton-NMR, GC, and GC/MS to provide detailed data on the components contained by this stream. Some of the GC data has been reported in the general analyses (Appendix D, Table D-1), providing concentrations of the quantities of phenol, cresols, xylenols, and other compounds present in the crude phenol. Such information is critical to the assessment of the economic value of the crude phenol since cresylic acids, particularly phenol, have been identified as highly valued products by prior marketing evaluations.[41] On the other hand, guaiacol (a methoxyphenol) and catechol (a diol isomer) have been identified as undesirable contaminants which would have to be removed in order to yield a marketable product.

With regards to the cresylic acids, the general analyses of the 1985-1987 data indicates that the crude phenol stream contains approximately 30.3 wt percent phenol, 21.6 wt percent cresols, 5.6 wt percent xylenols, and 2.0 wt percent ethylphenols. Elimination

of 1985 data from the statistical base changes these concentrations only slightly, if at all. This information is supplemented by Table 15, which provides some additional information on concentration-specific compounds such as o-, m-, and p-cresol, the various xylenol isomers, guaiacol, and catechol. This breakdown is of limited usefulness since it is extremely difficult to differentiate between some compounds and isomers. For example, most measurements lump meta- and para-cresol together. Ortho-, meta-, and para-ethylphenol are also lumped together, as are 2,4/2,5 xylenols and 2,3/2,6 xylenols. Furthermore, ethylphenol and 3,5 xylenol are in several instances measured as one peak on the GC and subsequently reported as equal concentrations, each comprising 50 percent of the measured concentration. Interpretation of the data is further clouded by significant differences between analyses by ANG, HRI, and an outside laboratory commissioned by ANG as illustrated by Table C-6. HRI's results are totally based on GC analysis while the ANG/Outside Lab utilized several different procedures in addition to GC analysis. Analyses reported by UNDEMRC appear to agree with that provided by HRI but is complicated by the fact that UNDEMRC reported their GC analyses on an area percent basis and HRI's is on a wt percent basis. While the response factors may not significantly shift the UNDEMRC data when converted to wt percent, this matter is not yet fully resolved. WRI has reported catechol and guaiacol concentrations of 4.26 wt percent and 1.47 wt percent respectively using GC/MS procedures. The need to arrive at an acceptable common understanding is highlighted by the disparity in reported phenol, guaiacol, and catechol concentrations.

UNDEMRC has completed a number of analyses identifying the types of compounds contained in the whole crude phenol as well as in specific cuts of the crude phenol. Both D-86 and TBP distillations have been used for this purpose. First, D-86 distillation fractions have been subjected to elemental analyses in order to provide a qualitative indication of the types of compounds present

as a function of boiling point. As can be seen from Table C-7, the nitrogen content tends to increase with boiling point while hydrogen and oxygen drop off slightly before increasing. However, all fractions of the crude phenol contain significant quantities of oxygen, as would be expected for this stream. Oxygen and hydrogen contents are markedly lower in the residue. Carbon content holds fairly constant, then declines between 60 percent and 94 percent distilled, but spikes upward in the residue, possibly as a result of regressive reactions taking place (i.e. decarboxylation and polymerization).

UNDEMRC also performed a rigorous analyses of TBP fractions cut from the crude phenol. A total of nine fractions were collected and analyzed using GC/MS and proton NMR. These fractions, and the quantity of material contained in each fraction is described in Figure B-5. The information in Tables C-8 and C-9 provide an indication of the functional groups present in the whole crude phenol and crude phenol fractions, respectively. GC/MS analyses presented in Table 10 provides a further indication of where various compounds are concentrated. For example, Fraction 1 contains mostly aniline, phenol, and some cresols. It also contains guaiacol, naphthalene, and several xylenol isomers, some in greater concentrations than several of the higher boiling fractions as a result of co-distillation with water in Fraction 1. Phenol is concentrated primarily in Fraction 3 (355^oF-356^oF) followed by Fractions 4 and 5 while the cresols tend to concentrate in Fractions 7 and 8. Such information can be helpful in determining where the crude phenol should be cut prior to any downstream processing.

Lastly, WRI has also performed a flash distillation of the crude phenol to prepare 450^oF^- and 450^oF^+ material for further upgrading experiments. The information collected for these fractions is summarized in Table C-11, while Figure B-6 illustrates the resulting distillation curves for the 450^oF^- and 450^oF^+ fractions.

8.3 Tar Oil

Tar oil is probably the most important of the GPGP by-product liquids since it represents the largest by-product stream. The tar oil also appears to be the most amenable to jet fuel production, while containing many potentially valuable compounds. Conversely, this material also poses some potentially troublesome processing problems due to the presence of heterocyclic and polycyclic compounds as well as solids carried over from the Lurgi gasifier. As a consequence, this stream has been subjected to more numerous and more detailed analyses than either the naphtha or crude phenol streams.

Statistical review of analytical data for the tar oil stream presented in Table 6 indicates that many of the commonly measured properties agree reasonably well over the entire 1985-1987 data base. The major exceptions to the statistical agreement involve viscosity, oxygen content, and GC component analyses. Examination of the statistical averages focusing only on that data of a more recent origin (12/1/86 - 12/31/87) yields similar agreement as illustrated in Table 7.

Subsequent comparison of the statistical averages in Tables 6 and 7 reveals some differences in properties have occurred over time, primarily in viscosity and elemental analyses. While the variation in viscosity is expected because of the relative lack of data, the difference in elemental analyses is not readily explained. One possibility results from the observation that the initial 20 vol percent of the tar oil appears to exhibit a trend toward lower boiling points over time. If some lighter, more hydrogen-rich material is being retained in the tar oil, then the H/C ratio would indeed increase. Another factor affecting both H/C ratio and viscosity may be thermal degradation, although that is not clear from the available information on sample handling. However, it has been observed that the tar oil can polymerize and

degrade during D-86 distillations. Consequently, it is possible that the tar oil analyses may be affected by the thermal history of a particular sample prior to testing. It is also possible that the presence of water and cresylic acids in the tar oil is interfering with O/N/S analysis. Overall, the more recent data should be chosen since it is representative of current operations.

Analyses of the D-86 distillation data presented in Tables 6 and 7 indicate that the distillation results show surprising agreement considering the degree of variation exhibited by several of the samples. Review of the 1985-87 and 1986-87 data base reveals that the range of data reported varies by as much as 150°F-175°F. However, general inspection of the D-86 data indicate that most distillation data are clustered in a relatively narrow band. When combined with the affect of the large sample population for most of the D-86 data, the statistical average and 95 percent confidence interval become insensitive to these few "outlayers".

As illustrated in Figures 11 and 12, the average D-86 distillation predicted from the collected data exhibits a tight 95 percent confidence interval. In fact, the 95 percent confidence level is generally within ± 5 percent of the average. The main exception to this is the first 10 percent and final 10 percent of the tar oil. For one thing, there is relatively little D-86 data available defining the last 10 percent of the tar oil. Observations by both WRI[32] and UNDEMRC[23] that the tar oil is thermally unstable explain why the D-86 procedure results in poor definition of the back end of the tar oil. It is also important to note that water present in the tar oil can interfere with analyses. In particular, WRI[32] observed that water caused frothing, resulting in a high IBP measurement. Changes in technique helped mitigate this problem during subsequent operations.

Review of statistical analyses data indicates that the standard deviation jumps from ~27°F for 50 percent distilled to 48°F at 70

percent distilled thus indicating that the data is showing much more variance over the last 20 to 30 LV percent of the tar oil. This observation has led several program participants to utilize distillation procedures which are specifically applicable to heavy hydrocarbon streams such as tar oil. Amoco, WRI, UNDEMRC, and DOD have performed ASTM D-1160 vacuum distillation and D-2887 GC distillation on a number of tar oil samples. Although there are significantly fewer D-1160 and D-2887 measurements available compared to the D-86 results, there are sufficient analyses to permit comparison of all three methods. First, the D-1160 and D-2887 data were analyzed statistically. Results presented in Table 8 indicate that while the D-1160 data appear to vary markedly, the D-2887 data agree quite well. However, the D-1160 results are significantly affected by the May 1985 Chromaspec analyses, while the other three measurements are much more recent. It is possible that the Chromaspec analyses reflects a real physical difference in the tar oil, an analytical error, or some other adjustment that has not been clearly identified. In any event, the Amoco and UNDEMRC data exhibit far better agreement once the Chromaspec data is dropped. Also, the low number of data points for the D-1160 and D-2887 distillations tends to result in a much higher level of statistical sensitivity than for the D-86 results.

The averaged D-2887 data were used as a basis for comparison. The D-86 and D-1160 results were then converted to a TBP distillation using standard procedures and compared to the D-2887 distillation. Comparison of the D-86, D-1160, and D-2887 data presented in Figure 12 indicate that the converted D-86 data agrees quite well with the D-2887 data up to about 70 percent distilled at which point the D-86 data falls apart. Similar inspection of the D-1160 and D-2887 data reveal the D-1160 consistently underpredicts the D-2887 and D-86 results. Therefore, the D-2887 distillation appears to be the preferred method for characterizing the entire tar oil stream. The D-86 is adequate for characterizing only the first 50-70 percent of the stream, while the D-1160 data requires

a closer review to determine why it underpredicts D-86 and D-2887 measurement.

Several miscellaneous distillation analyses have been conducted that are not directly comparable to the data discussed previously. For the most part, these data are aimed particularly at developing data and samples for subsequent processing tests. UNDEMRC has performed a simulated GC distillation (Table C-12) which reports the results in area percent, not weight or volume percent. Appropriate response factors must be applied to convert this information to either basis, a need which is highlighted by the comparison of the UNDEMRC simulated GC data and the average D-2887 data in Figure 25.

The University of Utah, Amoco and WRI have distilled the tar oil into several fractions and analyzed each fraction. These data are presented in Tables C-13, C-14, and C-15, respectively. The WRI data also includes analyses of a caustic-extracted tar oil sample, for which the D-2887 distillation is presented by Figure B-7. The University of Utah basically separated the fractions at 200°C (392°F) whereas WRI separated the tar oil at 450°F and 750°F, thus making comparison difficult. However, a look at the liquid chromatography and MS analyses from the University of Utah did identify a significant quantity of aliphatics in the 200°C$^-$ portion, and that both fractions contain significant amounts of hydroxyaromatics. The analyses also confirmed the higher concentration of polycyclic aromatic hydrocarbons and heterocyclics in the higher boiler range material.

As previously mentioned, WRI performed a caustic extraction on the tar oil in order to prepare material for hydrotreating tests. Although intent here was to reduce the oxygen content of the feedstock and thus reduce hydrogen consumption, this procedure can also recover potentially valuable cresylic acids for sale. The value of this approach is suggested by results from general com-

ponent analyses. These results, presented in Tables 6 and 7, have identified between 1.3-3.8 percent phenol, 3.1-8.1 percent cresol, and 2.4-5.8 percent xylenol in the tar oil. The possibility that the tar oil front end may contain cresylic acids is qualitatively verified by results of elemental analyses of D-86 fractions performed by UNDEMRC. It can be seen from the data presented in Table C-16 that the oxygen tends to concentrate in the 347°F-430°F fractions, which is fairly close to the boiling range for the cresylic acids (356°F-446°F). Furthermore, recent seasonal analyses by ANG[25] indicates that the phenol acid portion of the tar oil (obtained by neutral oil and tar base extraction) contains 9.5 percent phenol, 7.5 percent o-cresol, 18.1 percent m-and p-cresol, 9.0 percent 2,4 and 2.5 xylenol, 2.5 percent 2,3 and 2.6 xylenol, 13.8 percent 3,5 xylenol/ethyl phenols, and 1.7 percent guaiacol. These analyses have also indicated naphthalene may be present in concentrations ranging between 3.9-15.2 percent. According to J. Sinor,[41] naphthalene concentrations of 10 percent or higher could potentially justify recovery of naphthalene for sale as well as the cresylics.

As was the case with the Rectisol naphtha and crude phenol streams UNDEMRC has also performed detailed analyses of the tar oil using GC and GC/MS techniques, proton NMR analyses, and short-column chromatography. For example, the GC-FID data presented in Figure B-8 illustrate the multitude of compounds that are present in the tar oil while Table C-17 lists those compounds which have been identified. Although these data are reported in area percent and not weight percent, useful observations can be made regarding relative concentrations of particular components. For example, about 13 area percent of the tar oil is comprised of phenol, cresol, and xylenols, while another 13 area percent is toluene and xylene isomers. Another 8.5 area percent is naphthalenes, while approximately 13.4 area percent represents naphthalene isomers. Compounds such as acenaphthalene, biphenyl, fluorene, and pyrene have also been identified as have phenanthrene, various benzo-

furans, and aliphatics. This information is further supplemented by results of GC/MS, liquid-liquid extraction, and elution chromatography performed by WRI and presented in Tables C-18 and C-19. Comparison of results from liquid-liquid extractions by WRI and UNDEMRC indicate that the tar oil acids comprise between 20 and 28 wt percent of the total stream. Even though UNDEMRC has reported only 79 percent closure for their extraction, the tar acids would still represent about 22 percent of the tar oil even if the balance of material represented neutral oil and tar bases. Comparison of this information to ANG GC component analyses suggests that while the cresylic acid content of the tar oil acids is quite variable, and may range between 7 and 18 wt percent, it appears that most analyses lie between 7-10 percent. Consequently, it is concluded that the tar acids contain compounds other than phenol, cresols, and xylenols, a point verified by identification of naphthols and ethylphenols in the tar oil acids.

Basic nitrogen-containing compounds have been identified as well, using acid-base extraction followed by GC/MS analyses. As mentioned previously only 79 percent closure was obtained for the extraction and thus the data should be viewed as more of a qualitative, rather than a quantitative measurement of the types of compounds present. Furthermore, the tar oil bases represent only 2.5 wt percent of the total tar oil. However, well over 200 components were identified by the GC/MS and are listed in Table 3 of UNDEMRC's May 17, 1986 - August 16, 1987 Quarterly Technical Progress Report. As an illustration of the complex nature of this fraction, 35 isomers of both pyridine and quinoline were firmly identified along with 9 aniline isomers. Results of the GC/MS analysis as well as proton-NMR analyses are discussed in detail in the May 17, 1987 -August 16, 1987 UNDEMRC Quarterly Technical Progress Report.

It has been observed that the tar oil contains solids that potentially can result in downstream processing problems.

Laboratory characterization data suggests it can range between 0.01 and 0.1 wt percent of the tar oil, although Amoco has measured as much as 0.25 wt percent. WRI has experienced plugging of their reactor bed during upgrading tests and reports that the solids found in the reactor are representative of solids filtered from the feed. WRI observed that the solids are similar to that found in the earth's crust, and concluded that this material is probably from the coal ash.[32] Amoco's analysis indicates the particulates are fairly evenly spread between 4.7 to 19 microns.

9. CONCLUSIONS

This review and analysis of the GPGP liquid by-product data has identified a number of significant trends leading to the following conclusions:

- For tar oil, ASTM D-2887 distillation provides the most consistently reliable means of characterizing the entire boiling range of material - provided the appropriate standards are used.

- ASTM D-86 distillation procedure appear adequate for characterizing the first 95 percent of the naphtha and crude phenol streams and the first 80 percent of the tar oil.

- Thermal instability of the tar oil precludes use of the D-86 procedure beyond 80 percent recovery (600-650^{o}F) and may possibly result in substantial variations in stream properties (i.e, viscosity) with time.

- Specific gravity, elemental composition, and heating value data appear fairly reliable although heteroatoms (O,N,S) exhibit somewhat more variability, possibly due to water contained in samples or difficulty inherent in taking differences between small numbers (i.e., round-off).

- Water content, tar oil Conradson carbon, and GC component analyses are generally reliable with the exception of phenol and tar oil BTX/Lt Aromatics, methoxyphenol, diol- and naphthalene-isomer analyses.

- Naphtha aromatics content appears to be reliably predicted by PONA and GC component analyses. However, Paraffin/Olefin/Naphthene measurements vary significantly.

- General reliability of PONA analyses is weakened by infrequent testing, resulting in few data points separated by long periods of time.

- Crude phenol and tar oil viscosity data are highly variable with exception of tar oil viscosity at 150^{o}F.

- Although naphtha RVP appears to be reliable, data has not been collected since early 1986 and may not be representative.

- Naphtha produced through July 1985 differs from that produced subsequently as evidenced by consistent differences in RVP and D-86 IBP-30 percent characterization.

Finally, analysis of data collected before and after 1985 generally show little change with the exception of the naphtha and the tar oil cresylic acid concentrations. Also, the D-1160 tar oil distillation conducted by Chromaspec in 1985 differs markedly from more recent work-ups by UNDEMRC and Amoco. With exception of the Rectisol naphtha, it is likely that variations from continuing improvement of the analytical procedures is as important a cause of variability as any operational changes. Even in the case of the naphtha, handling procedures can significantly affect properties through the partial or complete loss of volatile compounds.

10. RECOMMENDATIONS

Overall, distillation, specific gravity, elemental composition, heating value, water content, tar oil Conradson carbon, and some GC component analysis appear to be reliable and can be utilized for process design, setting pilot plant operating conditions, and detailed equipment design. These are further supplemented by flash separation, GC-MS, proton- and C_{13}-NMR, and short column chromatography analyses conducted by WRI, UNDEMRC, Amoco, and others. Certain other data such methoxyphenols and diol-isomers, GC components analyses, and naphtha PONA are necessary for determining marketability and product value yet to establish a confident estimate for these purposes. It is up to the program participants to exercise proper judgement in utilizing these data. Since participants' understanding of these analytical procedures has improved and limitations identified as the program has proceeded, so has its' reliability. Consequently, it is recommended that the more recent data be used if a choice must be made. In any event, the data contained in this report should allow the user to evaluate particular data in the proper context.

Lastly, future characterization efforts should focus on acquiring a more consistent set of viscosity data, particularly at higher temperatures and also as a function of time and temperature. This is particularly important for the tar oil as it is thermally unstable and may have to be subjected to limits on storage temperature and/or time to minimize regressive reactions. Currently ongoing Amoco analytical studies should clarify whether or not this is a problem, and if so, what limitations must be observed. Naphtha PONA and RVP analyses would also benefit by further efforts, particularly before- and after-treating. Better definition of the quantities of methoxyphenols and diol-isomers is needed for the crude phenol and for the cresylic acid portion of the tar oil. Efforts by UNDEMRC, ANG, and HRI to quantify the crude phenol components as well as the products of HRI's Dynaphen process should provide valuable data in this area.

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FIGURES
and
TABLES

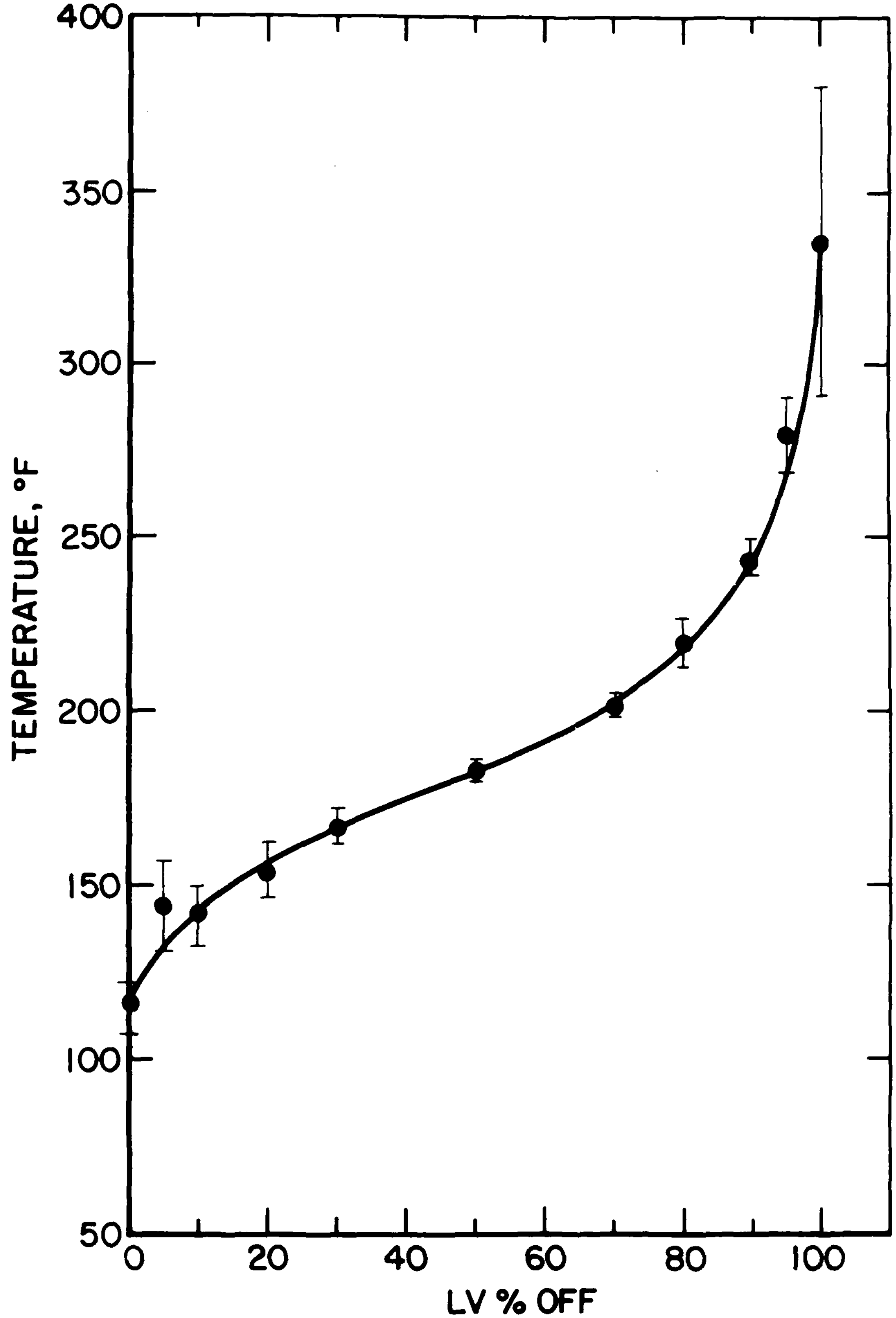

Figure 1. Average D-86 Rectisol Naphtha Distillation. (includes all 1985-87 data)

NO./16.358

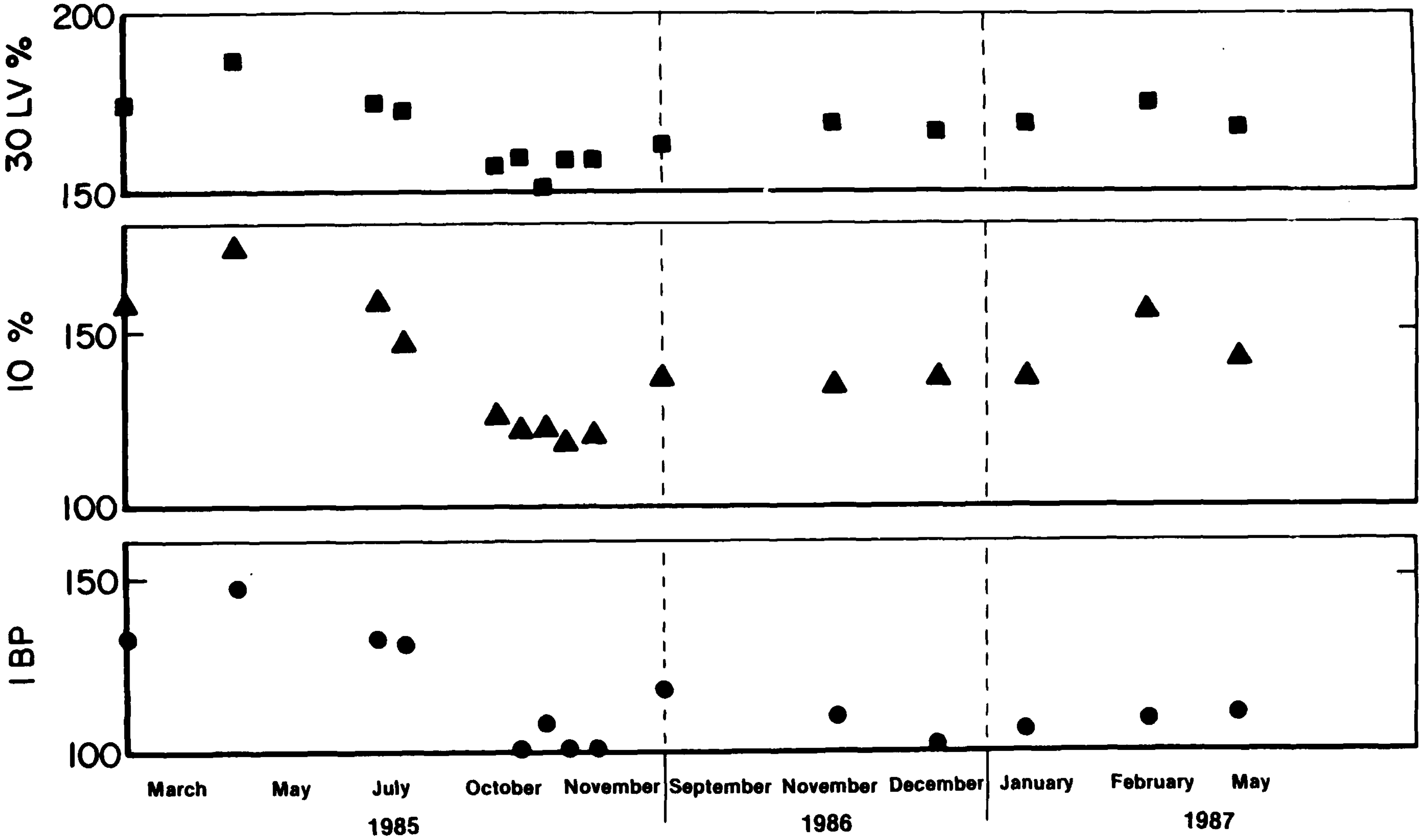

Figure 2. Rectisol Naphtha Boiling Point History.

NO/16,359

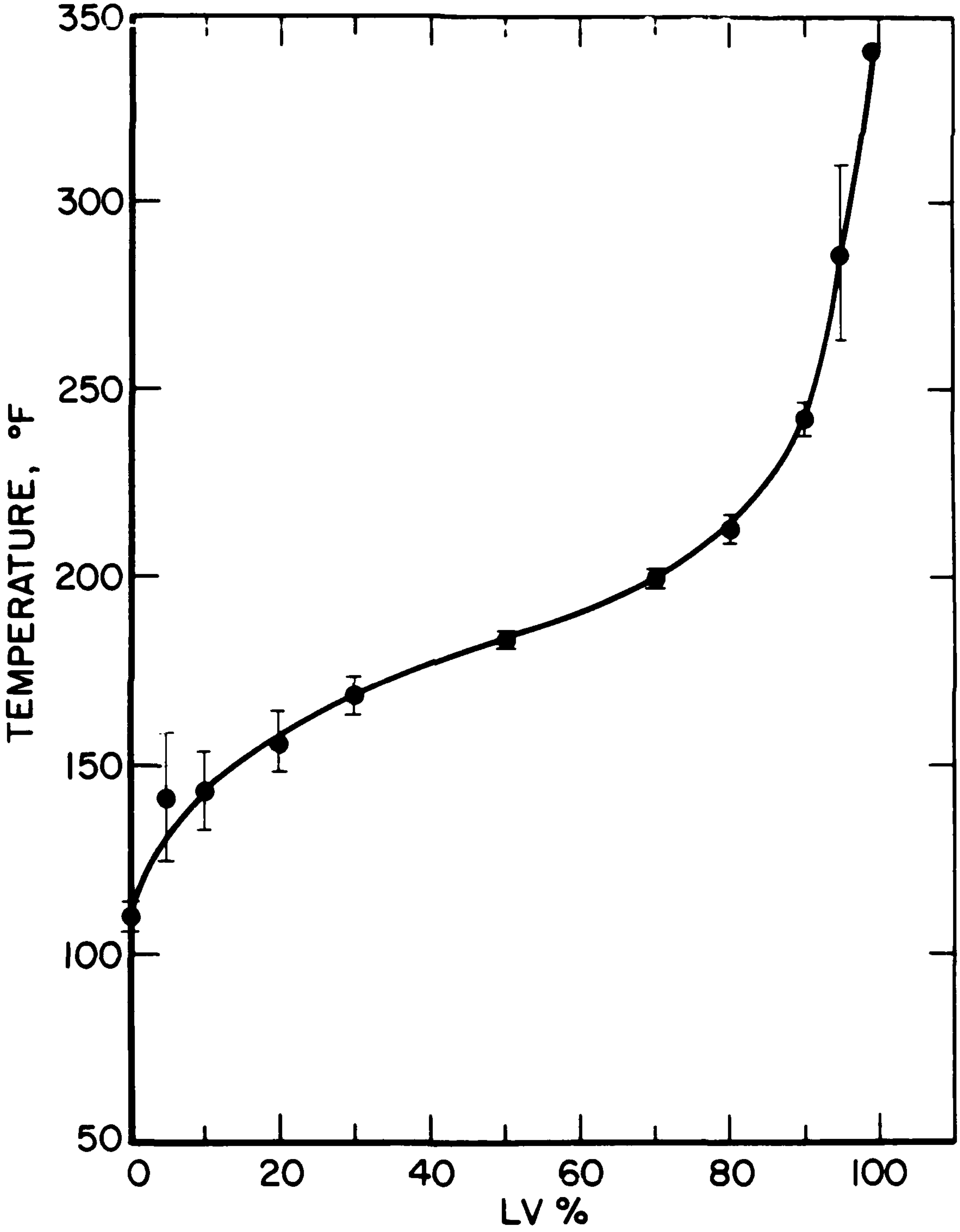

Figure 3. D-86 Rectisol Naphtha Distillation (excluding 1985 Data).

NO./16.360

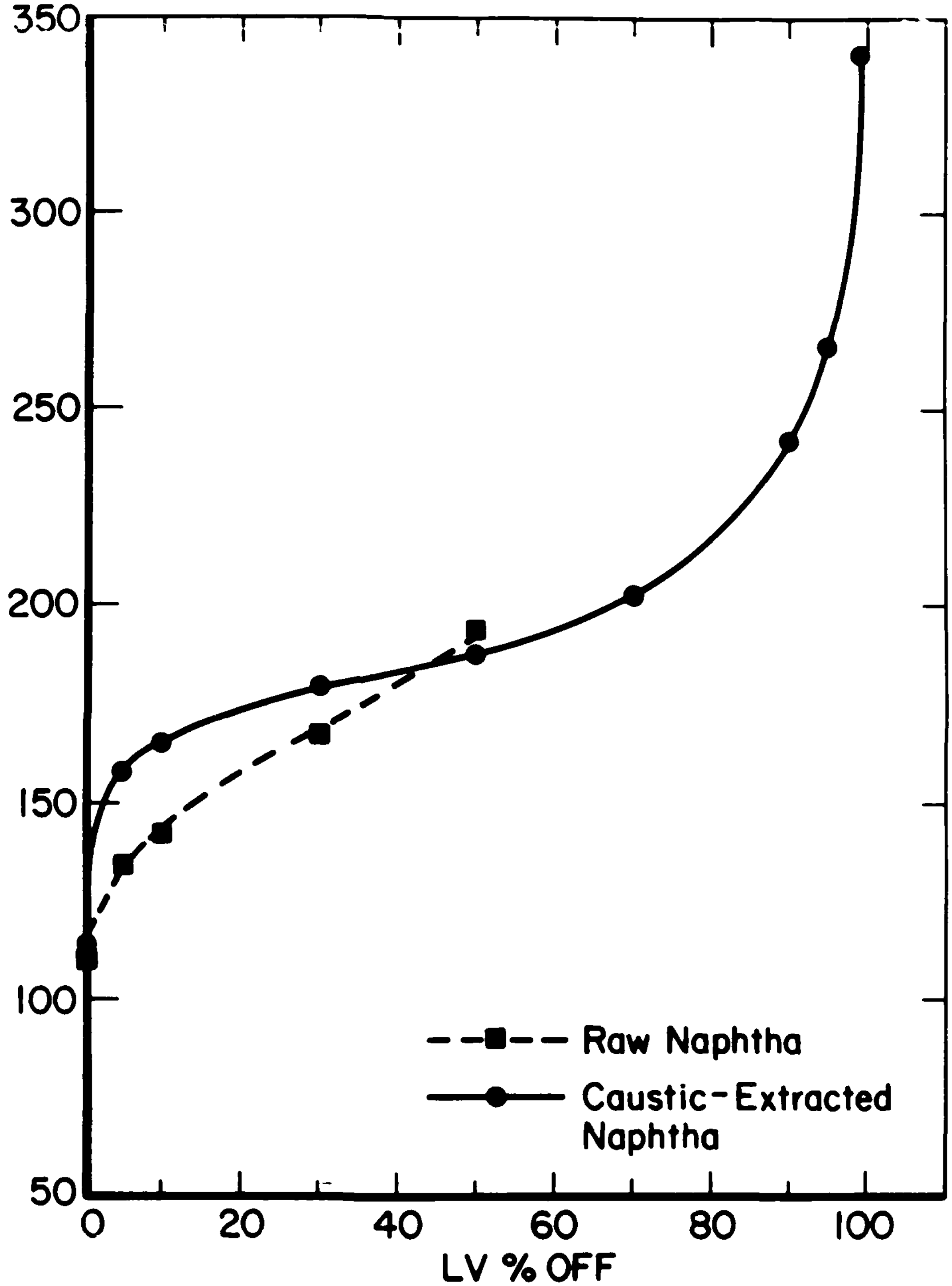

Figure 4. Comparison of Amoco D-86 for Raw- and Caustic-Extracted Rectisol Naphtha.

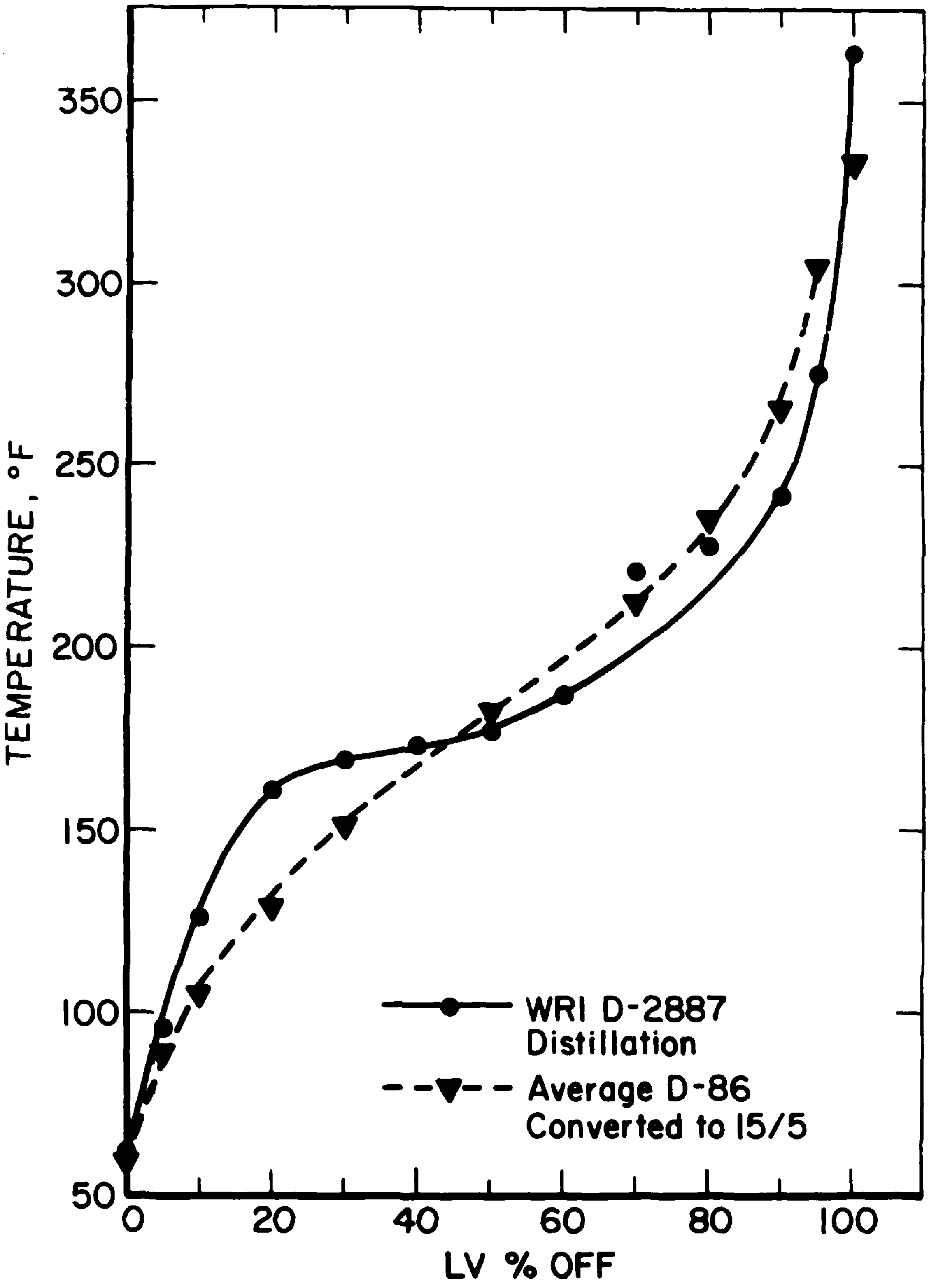

Figure 5. Comparison of Average and WRI Rectisol Naphtha Distillation.

NO./16,362

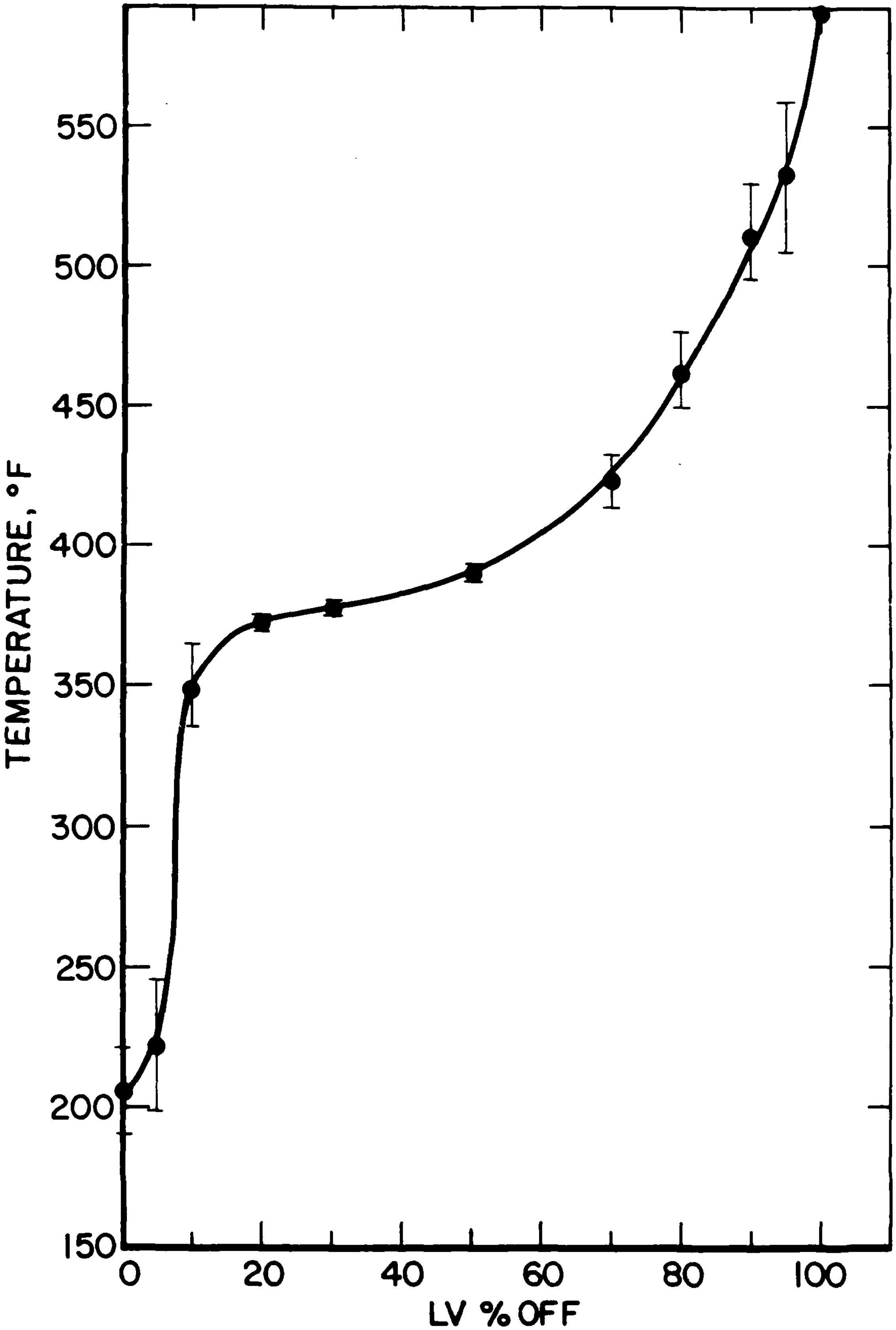

Figure 6. Crude Phenol D-86 Distillation.
(including 1985 data)

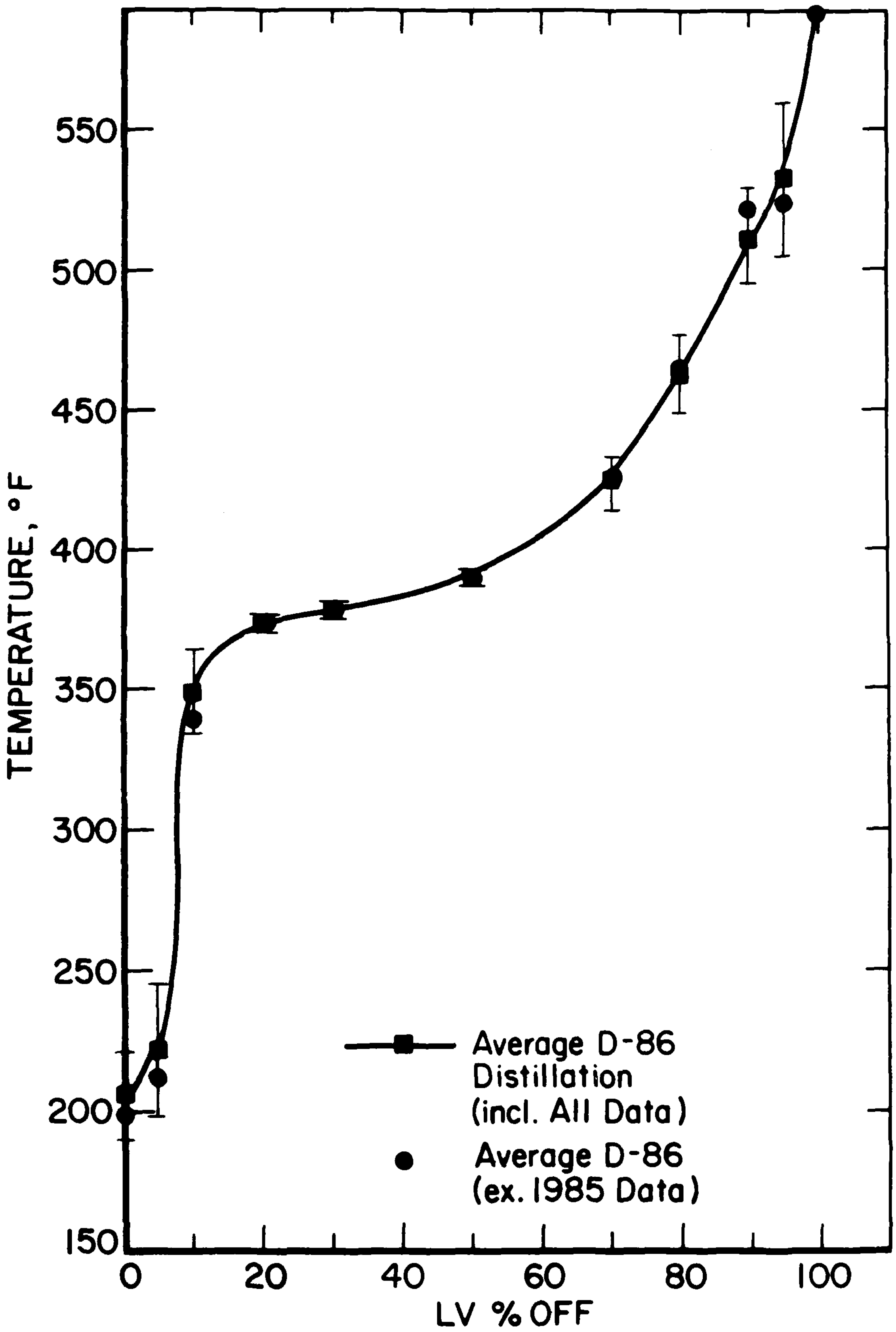

Figure 7. Crude Phenol D-86 Distillation (excluding 1985 Data).

NO.: 16.364

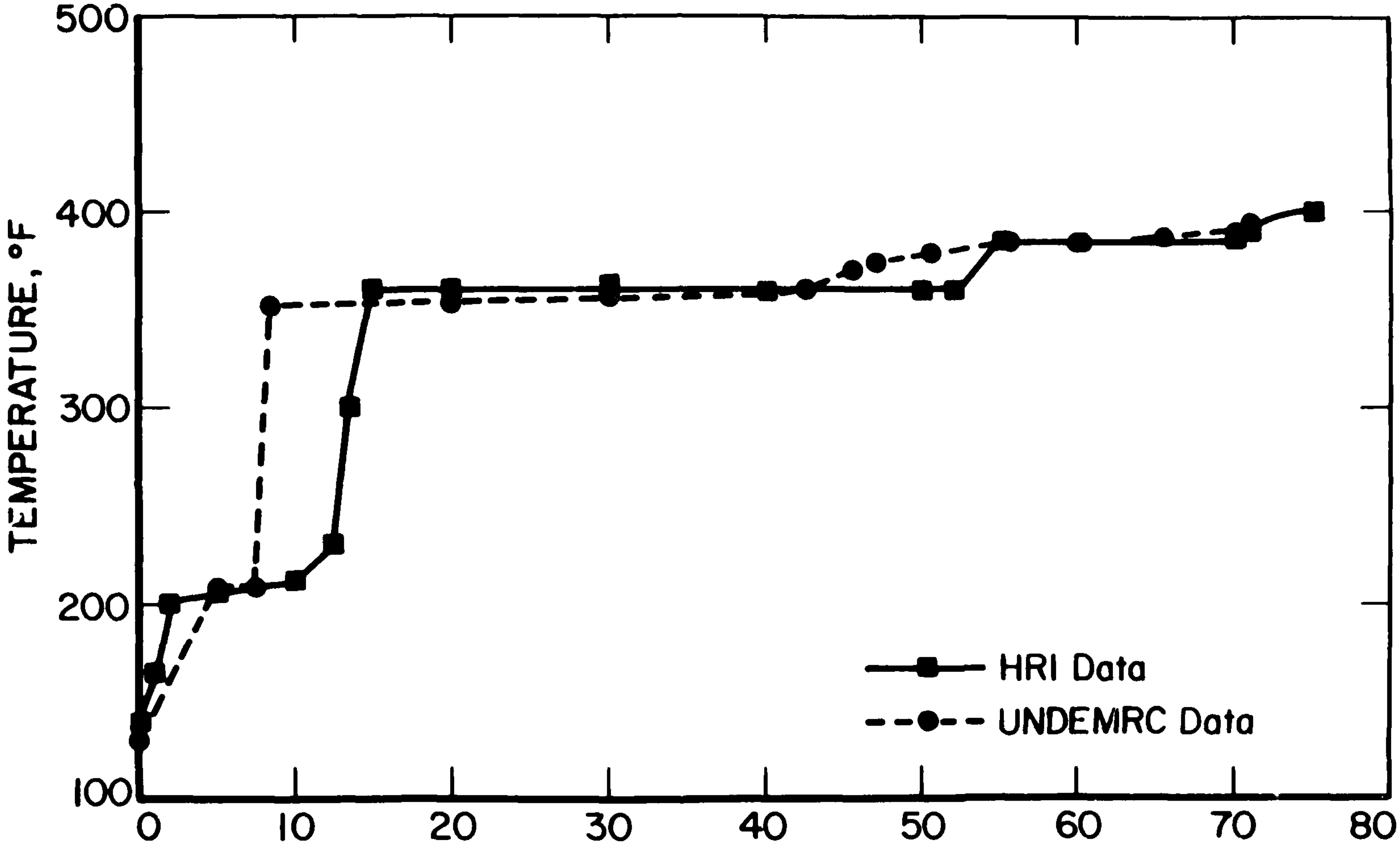

Figure 8. Comparison of UNDEMRC and HRI Crude Phenol TBP Distillation.

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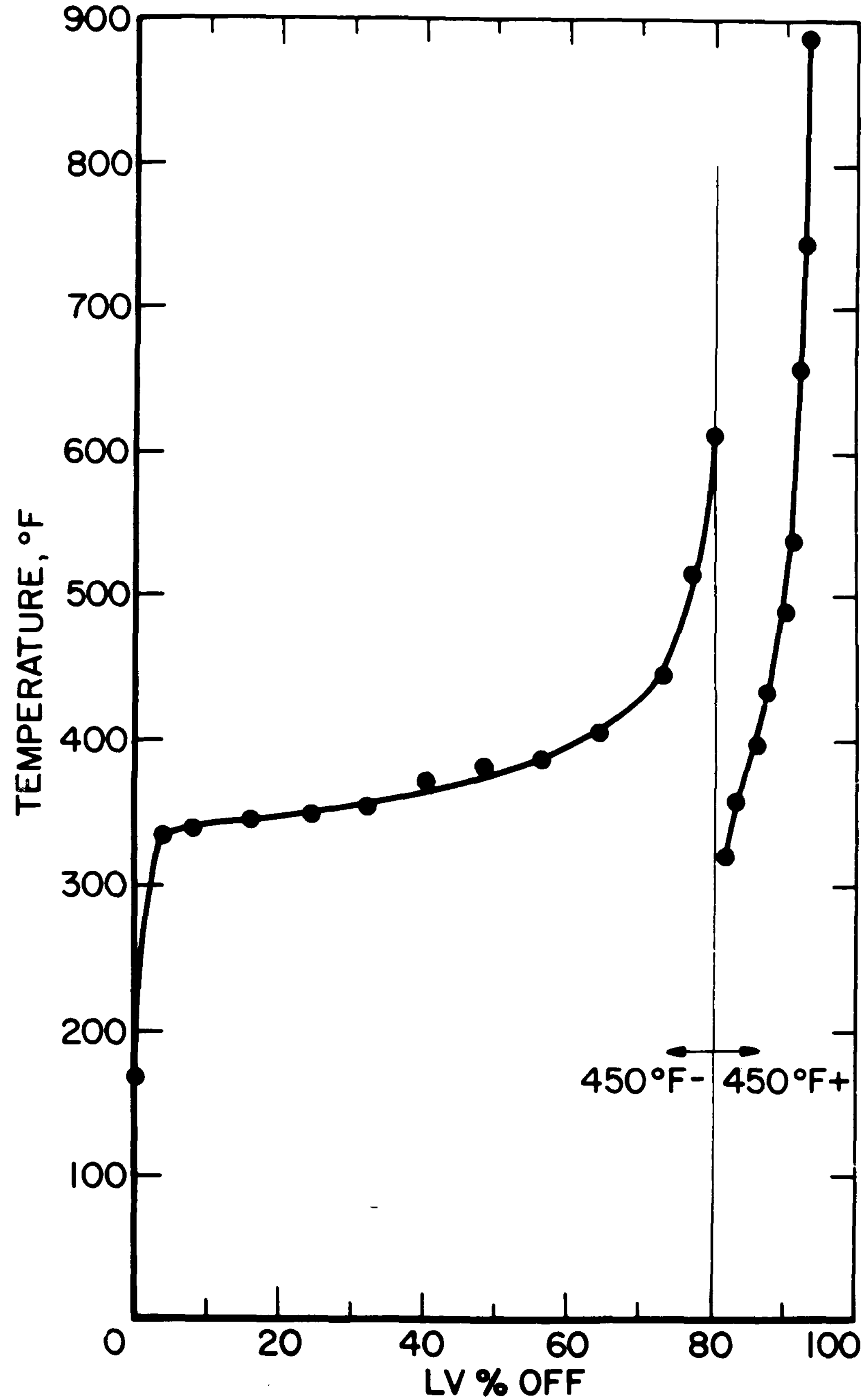


Figure 9. WRI D-2887 Analysis of Crude Phenol 450°F Flash Separation Fractions.

NO./16,366

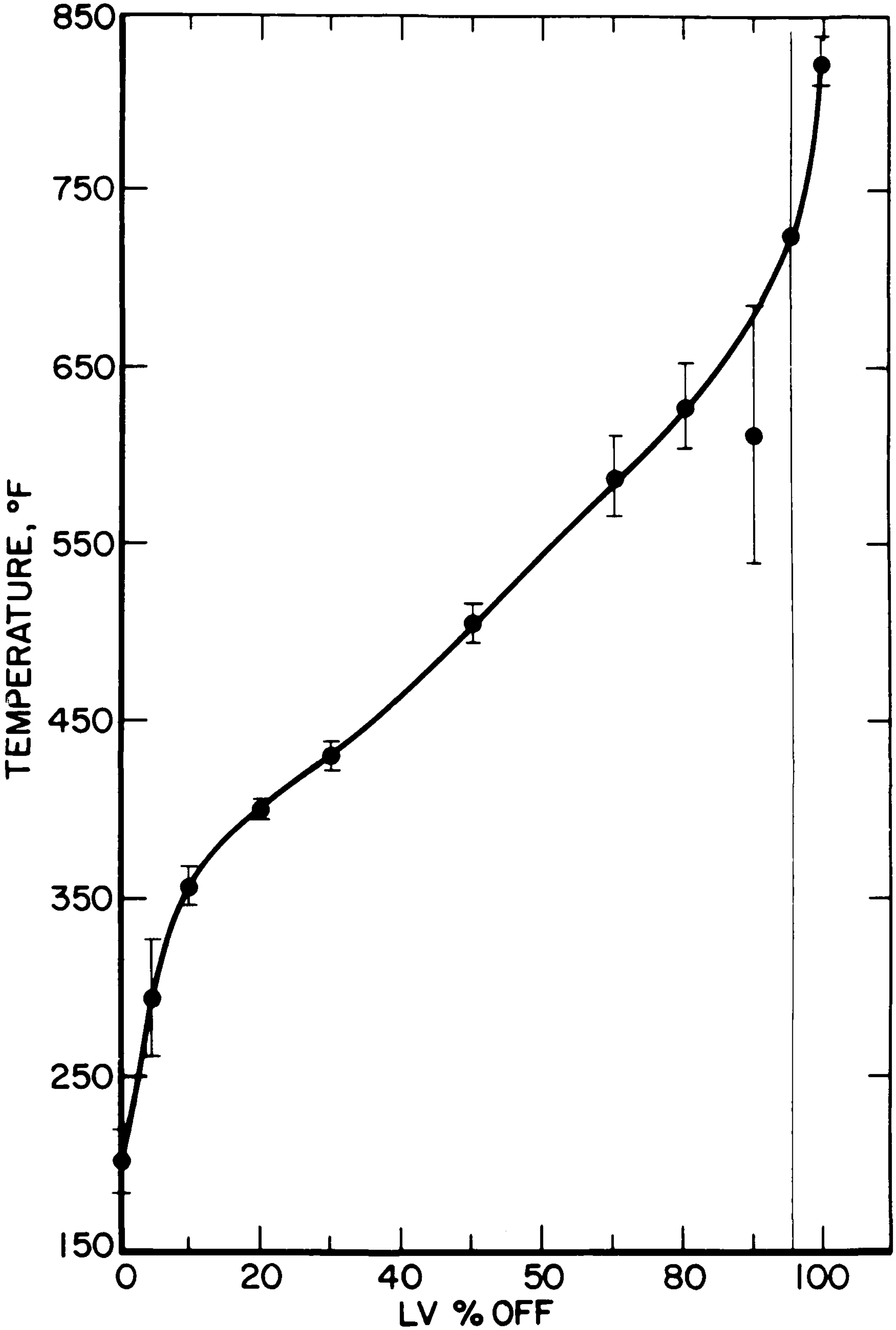


Figure 10. Average D-86 Tar Oil Distillation (including 1985 data)

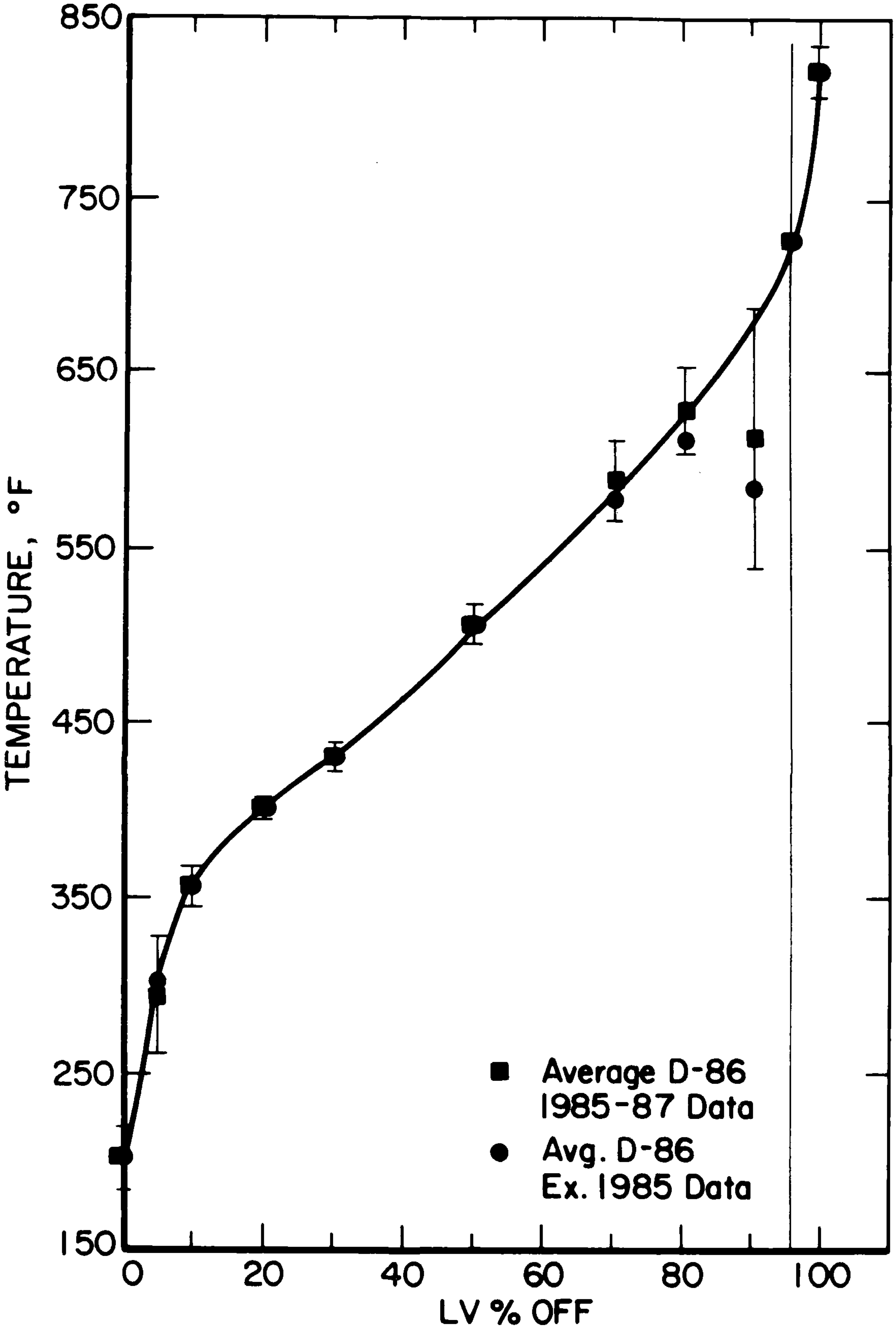


Figure 11. D-86 Tar Oil Distillation excluding 1985 Data.

O./16,368

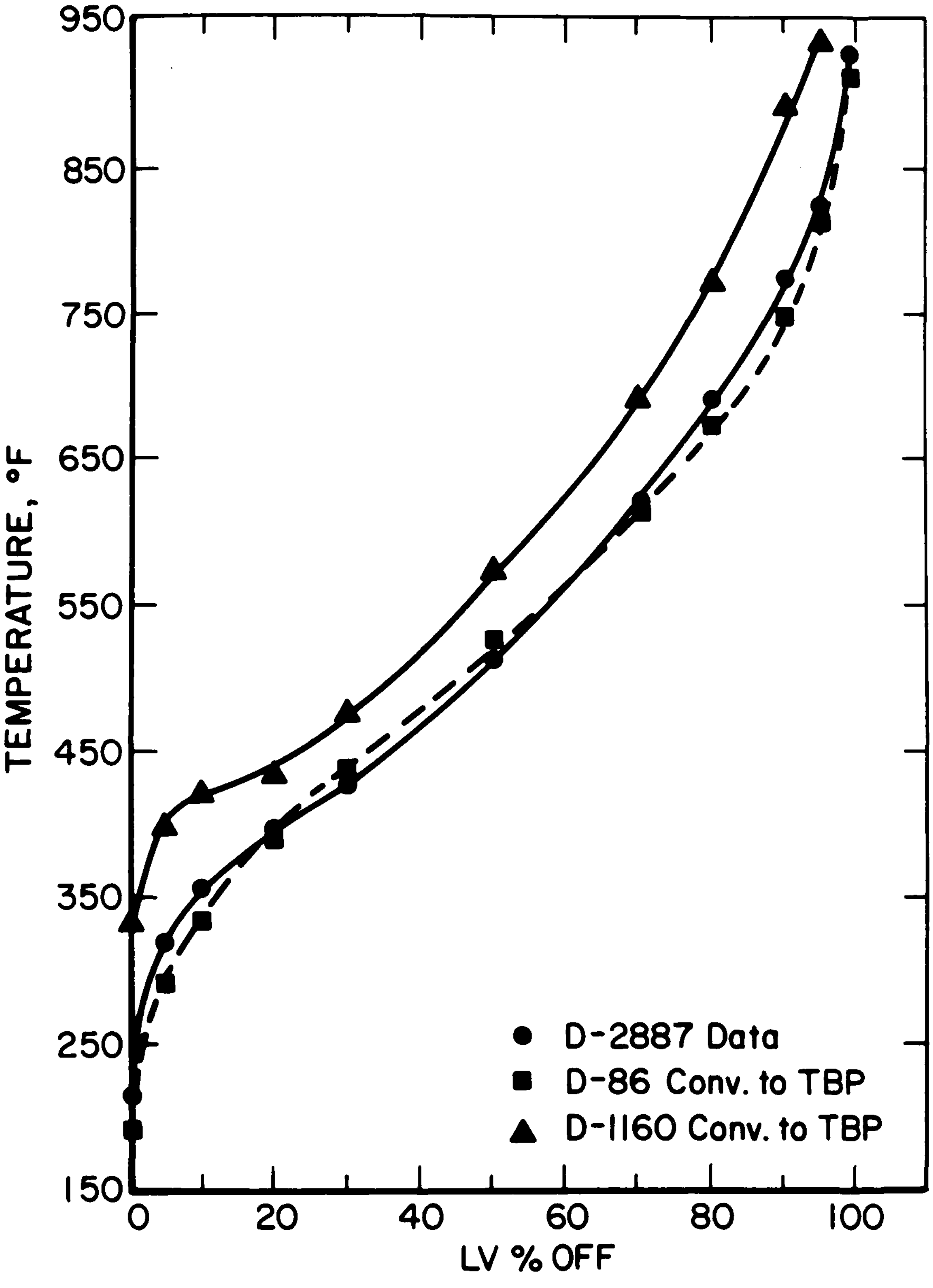


Figure 12. Comparison of D-86, D-2887, and D-1160 Tar Oil Distillation Data.

NO./16.369

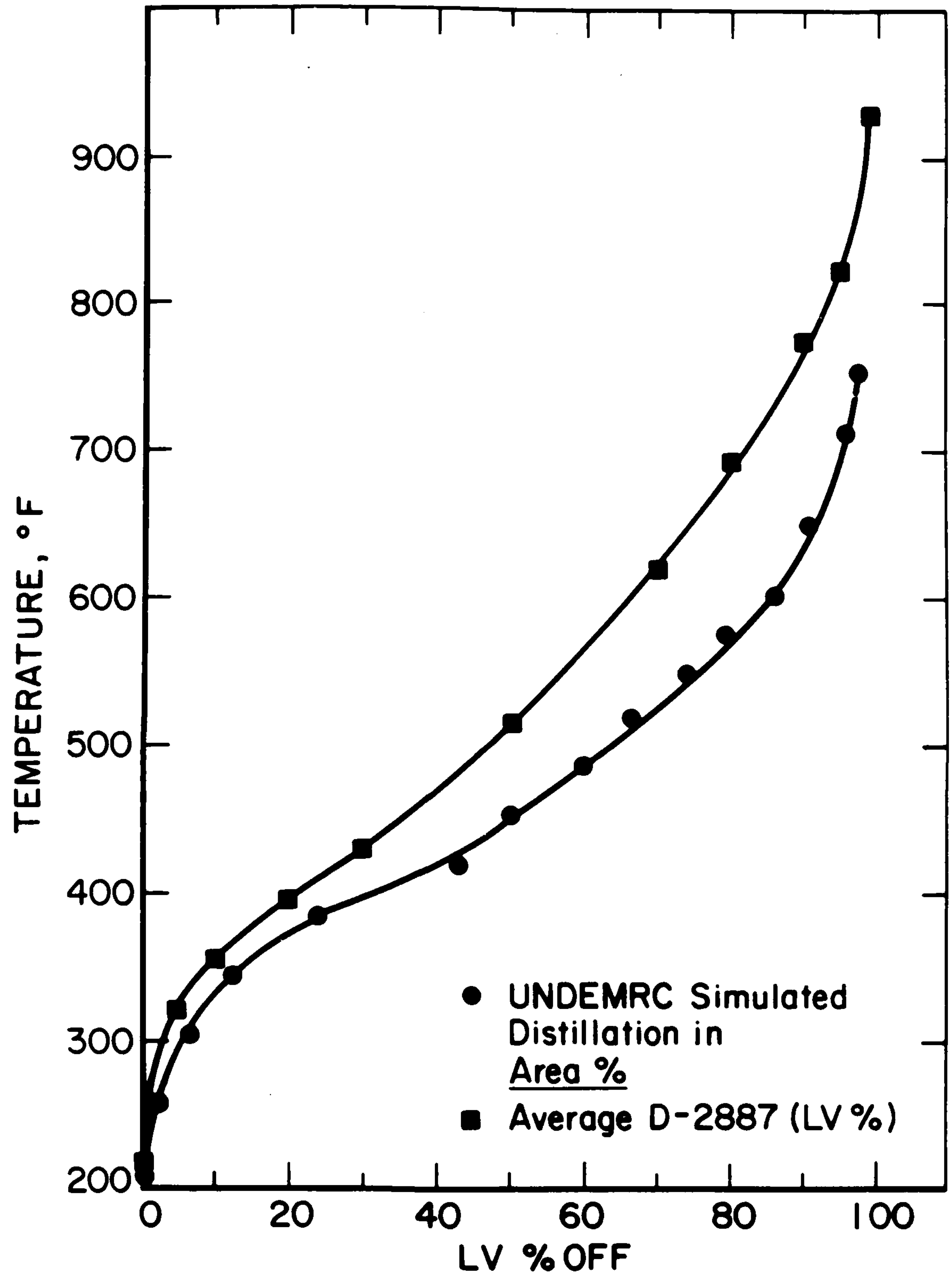


Figure 13. Comparison of D-2887 Tar Oil Distillation to UNDEMRC GCD.

Table 1.
Statistical Analyses of GPGP Rectisol Naphtha Characterization Data

		sample size	AVERAGE	95% Confidence Limit	95% Confidence Limit	Range of Reported Data MIN	MAX
Distillation (ASTM D-86)	LV %		(deg F)	(+/-)	(% of mean)		
	IBP	16	114	7	6.4	100	147
	5	9	143	11	7.5	117	170
	10	17	141	8	6.0	118	174
	20	14	156	8	4.8	138	180
	30	17	167	5	2.8	150	186
	50	17	183	3	1.7	170	197
	70	15	202	3	1.6	190	214
	80	15	220	6	2.6	210	244
	90	16	247	5	2.2	232	270
	95	14	281	11	4.0	244	335
	99	1	331	0	0.0	331	331
	% REC	11	98	0.9	0.9	94	99.5
Specific Gravity	60/60 F	18	0.825	0.004	0.5	0.806	0.843
	API	18	40.0	0.8	2.0	36.4	44.0
Vapor Pressure	RVP	7	8.3	2.4	28.8	3.9	11.0
Heating Value(HHV)	Btu/lb	13	17035	166	1.0	16420	17500
Water Content	Wt%	14	0.52	0.11	21.8	0.20	0.80
Elemental Analysis	Wt%-Dry						
	C	14	85.8	0.9	1.1	83.8	89.8
	H	14	9.4	0.4	4.5	8.2	11.0
(By Difference)	O	14	3.1	0.8	26.2	0.1	5.4
	N	14	0.3	0.1	41.3	0.1	0.8
	S	14	1.3	0.3	19.4	0.5	2.3
	Sum	14	99.9	0.1	0.1	99.0	100.0
Atomic H/C Ratio		14	1.32	0.07	4.9	1.14	1.56
PONA	LV%						
-Paraffins		4	14.8	7.4	49.9	8.4	21.5
-Olefins		3	10.7	12.1	112.4	4.1	15.6
-Naphthenes		4	11.9	6.4	53.6	5.4	16.1
-Aromatics		4	63.5	3.4	5.3	60.0	65.5
Component Analysis	Wt%						
Methanol		14	2.0	0.8	37.4	0.3	5.9
Acetone		13	5.6	1.1	19.7	3.2	9.1
MEK		13	3.2	0.7	21.7	1.8	5.0
Benzene		9	42.4	3.0	7.2	37.3	50.3
Toluene		9	15.9	1.5	9.7	13.9	20.5
Xylene		9	3.6	0.7	19.9	2.4	6.0
Ethyl Benzene		1	0.9	**********	**********	0.9	0.9
Phenolics		1	4.7	**********	**********	4.7	4.7
Total BTX		9	61.9	5.0	8.1	54.5	76.8

Table 2.
Statistical Analysis of GPGP Rectisol Naphtha Characterization Data (excluding data through 7/19/85)

		sample size	AVERAGE (deg F)	95% Confidence Limit	95% Confidence Limit	Range of Reported Data MIN	MAX
Distillation (ASTM D-86)	LV %			(+/-)	(%)		
	IBP	12	107	4	3.6	100	118
	5	6	137	11	8.3	117	149
	10	13	136	9	6.3	118	162
	20	11	153	8	5.2	138	171
	30	13	165	5	3.0	150	178
	50	13	181	3	1.7	170	189
	70	11	200	4	1.8	190	212
	80	11	218	6	2.7	210	243
	90	12	245	6	2.4	232	270
	95	10	278	16	5.7	244	335
	99	0	----	n.a.	n.a.	----	----
	% REC	9	98	1.1	1.1	94	99.5
Specific Gravity	60/60 F	14	0.823	0.004	0.4	0.806	0.832
	API	14	40.5	0.8	1.9	38.6	44.0
Vapor Pressure	RVP	5	9.8	1.2	12.4	8.0	11.0
Heating Value(HHV)	Btu/lb	9	17048	159	0.9	16500	17200
Water Content	Wt%	10	0.61	0.09	14.3	0.40	0.80
Elemental Analysis	Wt%-Dry						
	C	12	85.4	0.7	0.9	83.8	87.6
	H	12	9.4	0.5	5.2	8.2	11.0
(By Difference)	O	12	3.5	0.8	21.6	1.0	5.4
	N	12	0.3	0.1	44.5	0.1	0.8
	S	12	1.4	0.3	21.3	0.5	2.3
	Sum	12	99.9	0.2	0.2	99.0	100.0
Atomic H/C Ratio		12	1.32	0.07	5.6	1.14	1.56
PONA	LV%						
-Paraffins		2	11.7	29.6	253.4	8.4	15.0
-Olefins		2	14.1	13.9	99.1	12.5	15.6
-Naphthenes		2	8.8	30.1	344.0	5.4	12.1
-Aromatics		2	62.0	17.5	28.3	60.0	63.9
Component Analysis	Wt%						
Methanol		12	1.8	0.4	23.1	0.3	3.0
Acetone		11	5.4	1.2	22.1	3.2	9.1
MEK		11	3.1	0.7	22.7	1.8	5.0
Benzene		8	41.4	2.5	6.0	37.3	46.2
Toluene		8	15.4	1.1	7.0	13.9	18.0
Xylene		8	3.3	0.3	9.2	2.4	3.6
Ethyl Benzene		1	0.9	n.a.	n.a.	0.9	0.9
Phenolics		0	----	----	----	----	----
Total BTX		8	60.0	3.4	5.7	54.5	67.2

Table 3.
Statistical Analyses of GPGP Rectisol Naphtha Characterization Data (excluding 1985 data)

		sample size	AVERAGE (deg F)	95% Confidence Limit	95% Confidence Limit	Range of Reported Data MIN	MAX
Distillation (ASTM D-86)	LV %			(+/-)	(%)		
	IBP	8	110	4	4.1	102	118
	5	5	141	9	6.1	131	149
	10	8	145	9	6.1	134	162
	20	7	160	8	4.9	149	171
	30	8	170	4	2.5	162	178
	50	8	185	2	1.1	182	189
	70	7	201	3	1.3	198	207
	80	7	216	4	1.7	210	221
	90	7	248	10	3.9	236	270
	95	5	291	28	9.7	270	335
	99	0	n.a.	n.a.	n.a.	----	----
	% REC	4	98	1.6	1.7	97	99.5
Specific Gravity	60/60 F	9	0.824	0.004	0.5	0.816	0.832
	API	9	40.3	0.8	2.1	38.6	42.0
Vapor Pressure	RVP	0	n.a.	n.a.	n.a.	----	----
Heating Value (HHV)	Btu/lb	5	16946	284	1.7	16500	17130
Water Content	Wt%	5	0.54	0.13	23.4	0.40	0.70
Elemental Analysis	Wt%-Dry						
	C	7	84.6	0.5	0.6	83.8	85.4
	H	7	9.7	0.7	6.8	8.7	11.0
(By Difference)	O	7	4.1	0.8	18.9	2.9	5.4
	N	7	0.3	0.2	73.4	0.1	0.8
	S	7	1.2	0.4	34.5	0.5	1.8
	Sum	7	99.9	0.3	0.3	99.0	100.0
Atomic H/C Ratio		7	1.37	0.10	7.0	1.23	1.56
PONA	LV%						
-Paraffins		2	11.7	29.6	253.4	8.4	15.0
-Olefins		2	14.1	13.9	99.1	12.5	15.6
-Naphthenes		2	8.8	30.1	344.0	5.4	12.1
-Aromatics		2	62.0	17.5	28.3	60.0	63.9
Component Analysis	Wt%						
Methanol		7	1.6	0.6	39.7	0.3	2.5
Acetone		6	5.9	2.1	35.9	3.2	9.1
MEK		6	3.1	1.1	36.7	1.8	4.6
Benzene		7	42.0	2.5	6.0	39.0	46.2
Toluene		7	15.6	1.2	7.4	13.9	18.0
Xylene		7	3.3	0.4	10.9	2.4	3.6
Ethyl Benzene		1	0.9	**********	**********	0.9	0.9
Phenolics		0	n.a.	n.a.	n.a.	----	----
Total BTX		7	60.8	3.5	5.7	57.2	67.2

Table 4.
Statistical Analysis of GPGP Crude Phenol Characterization Data

		sample size	AVERAGE (deg F)	95% Confidence Limit	95% Confidence Limit	Range of Reported Data MIN	MAX
Distillation (ASTM D-86)	LV %			(+/-)	(%)		
	IBP	16	206	16	7.6	170	311
	5	5	222	24	10.7	205	258
	10	16	349	15	4.3	267	374
	20	16	373	3	0.7	365	381
	30	16	378	2	0.7	367	384
	50	16	390	3	0.8	375	400
	70	16	424	9	2.2	390	466
	80	16	463	14	3.0	392	520
	90	16	512	17	3.3	457	600
	95	5	533	27	5.0	509	571
	99	1	592	0	0.0	592	592
	% REC	13	93	1	1.4	90	96
Specific Gravity	60/60 F	18	1.067	0.003	0.305	1.056	1.080
	API	18	1.1	0.4	36.6	-0.5	2.5
Vapor Pressure	RVP	0	----	----	----	----	----
Heating Value(HHV)	Btu/lb	10	14057	354	2.5	13520	14720
Water Content	Wt%	17	4.4	0.6	13.8	1.9	5.5
Elemental Analysis	Wt%-Dry						
	C	15	77.4	1.1	1.4	75.2	82.4
	H	15	7.5	0.3	4.2	6.5	8.7
(By Difference)	O	15	14.5	1.1	7.7	10.1	17.1
	N	15	0.4	0.1	16.4	0.2	0.7
	S	15	0.1	0.0	31.2	0.0	0.3
	Sum	15	100.0	0.1	0.1	99.6	100.1
Atomic H/C Ratio		15	1.2	0.1	4.4	1.0	1.3
Viscosity							
-@ 80 F	SUS	7	171.8	156.9	91.3	8.4	530.0
100 F		4	110.7	194.3	175.6	15.6	320.0
212 F		2	19.5	94.3	483.8	9.0	30.0
-@ 100 F	cSt	4	65	59	91.0	8.2	112
210 F		1	2	0	0.0	1.5	1.5
Component Analysis	Wt%						
BTX/Lt. Aromatics		1	0.4	0.0	0.0	0.4	0.4
Phenols		7	30.3	3.6	11.8	23.8	36.6
Cresols		7	21.6	2.6	11.9	17.0	25.2
Methoxyphenol		3	1.2	0.2	17.5	1.0	1.3
Ethylphenol		5	2.0	0.2	11.3	1.8	2.2
Xylenols		6	5.6	1.6	29.6	4.3	9.0
Naphthalene		0	----	----	----	----	----
Diols isomers		3	1.2	1.8	148.3	0.3	2.1

Table 5.
Comparison of GPGP Crude Phenol Analyses w/ & w/o 1985 Data

		1985-1987 Data			ex 1985 Data		
		data points	AVERAGE (w/ 95% Conf. lim.)		data points	AVERAGE (w/ 95% Conf. lim.)	
Distillation (F) (ASTM D-86)	LV %						
	IBP	16	206 +/-	16	10	198 +/-	9
	5	5	222	24	4	213	10
	10	16	349	15	10	339	22
	20	16	373	3	10	372	3
	30	16	378	2	10	377	3
	50	16	390	3	10	389	3
	70	16	424	9	10	425	14
	80	16	463	14	10	466	17
	90	16	512	17	10	523	22
	95	5	533	27	4	524	18
	99	1	592	0	0	----	----
	% REC	13	93	1	8	93 +/-	2
Specific Gravity	60/60 F	18	1.067 +/-	0.003	12	1.065 +/-	0.00
	API	18	1.1	0.4	12	1.4	0.46
Vapor Pressure	RVP	0	----	----	0	----	----
Heating Value(HHV)	Btu/lb	10	14057 +/-	354	7	14039 +/-	502
Water Content	Wt%	17	4.4 +/-	0.6	11	4.4 +/-	0.9
Elemental Analysis	Wt%-Dry						
C		15	77.4 +/-	1.1	12	76.9 +/-	0.8
H		15	7.5	0.3	12	7.6	0.3
(By Difference) O		15	14.5	1.1	12	14.8	1.0
N		15	0.4	0.1	12	0.4	0.1
S		15	0.1	0.0	12	0.1	0.0
	Sum	15	100.0	0.1	12	100.0	0.1
Atomic H/C Ratio		15	1.2	0.1	12	1.19 +/-	0.05
Viscosity							
-@ 80 F	SUS	7	171.8 +/-	156.9	5	124.3 +/-	120.5
100 F		4	110.7	194.3	1	15.6	----
212 F		2	19.5	94.3	0	----	----
-@ 100 F	cSt	4	65	59	2	70.5 +/-	58.8
210 F		1	2	0	0	----	----
Component Analysis	Wt%						
BTX/Lt. Aromatics		1	0.4 +/-	0.0	1	0.4 +/-	0.0
Phenols		7	30.3	3.6	6	30.0	4.3
Cresols		7	21.6	2.6	6	21.1	2.8
Methoxyphenol		3	1.2	0.2	3	1.2	0.2
Ethylphenol		5	2.0	0.2	5	2.0	0.2
Xylenols		6	5.6	1.6	6	5.6	1.6
Naphthalene		0	----	----	0	----	----
Diols isomers		3	1.2	1.8	3	1.2	1.8

Table 6.
Statistical Analysis of GPGP Tar Oil Characterization Data

		sample size	AVERAGE	95% Confidence Limit	95% Confidence Limit	Range of Reported Data MIN	MAX
Distillation (F) (ASTM D-86)	LV %			(+/-)	(%)		
	IBP	24	199	14	7.2	150	320
	5	11	297	30	10.0	227	371
	10	26	357	10	2.7	258	388
	20	26	401	6	1.4	363	425
	30	26	432	7	1.6	385	460
	50	26	509	11	2.1	424	550
	70	23	589	21	3.6	480	655
	80	20	627	24	3.8	525	700
	90	7	619	58	9.3	565	728
	95	2	696	858	123.4	600	791
	99	1	824	0	0.0	824	824
	% REC	25	81	5	5.6	60	99
Specific Gravity	60/60 F	23	1.017	0.003	0.29	0.996	1.029
	API	23	7.7	0.4	5.2	6.0	10.5
Viscosity	SUS						
-70 F		6	608	342	56.2	260	1100
-80 F		5	1084	462	42.6	530	1530
-100 F		3	766	1700	221.8	79	1700
-120 F		9	107	64	59.7	53	320
-150 F		4	66	8	12.2	60	74
-175 F		1	40	0	0.0	40	40
Water Content	Wt%	20	2.1	0.2	10.1	1.5	3.4
Elemental Analysis	Wt%-Dry						
	C	17	86.1	1.4	1.7	82.6	91.0
	H	17	8.8	0.2	2.8	7.7	9.7
(By Difference)	O	16	4.0	1.3	33.2	0.0	6.9
	N	19	0.6	0.1	15.6	0.4	1.0
	S	18	0.5	0.0	9.8	0.3	0.7
	Sum	16	100.1	0.1	0.1	99.9	100.8
Atomic H/C Ratio		17	1.2	0.0	3.7	1.1	1.4
Heating Value(HHV)	Btu/lb	18	16483.3	69.2	0.4	16200.0	16900.0
Gross Component Analysis	Wt%						
BTX/Lt Aromatics		4	5.1	3.9	76.8	1.8	7.6
Phenols		6	1.9	0.9	47.5	1.3	3.8
Cresols		6	4.5	1.7	38.9	3.1	8.1
Methoxyphenols		2	0.5	0.4	99.8	0.4	0.5
Ethylphenol		6	1.0	0.3	33.0	0.7	1.7
Xylenol		6	3.3	1.2	37.2	2.4	5.8
Naphthalene Isomers		3	7.9	12.8	161.2	3.9	15.2
CCR	Wt%	6	3.3	0.4	13.4	2.6	4.0

Table 7.
Statistical Analysis of GPGP Tar Oil Characterization Data (excluding 1985 data)

		sample size	AVERAGE	95% Confidence Limit	95% Confidence Limit	Range of Reported Data MIN	MAX
Distillation (ASTM D-86)	LV %			(+/-)	(%)		
	IBP	18	202	18	9.0	166	320
	5	8	300	36	12.1	227	365
	10	18	357	14	3.9	258	388
	20	18	403	7	1.7	381	425
	30	18	433	8	1.8	405	460
	50	18	510	10	2.0	473	550
	70	15	583	17	2.9	511	630
	80	12	607	24	3.9	541	670
	90	6	620	61	9.8	565	737
	95	2	696	858	123.4	600	791
	99	1	824	0	0.0	824	824
	% REC	17	79	7	8.2	60	99
Specific Gravity	60/60 F	15	1.018	0.003	0.33	1.007	1.029
	API	15	7.5	0.5	6.1	6.0	9.0
Viscosity	SUS						
-70 F		3	920	316	34.4	830	1100
-80 F		5	1084	462	42.6	530	1530
-100 F		0	ERR	ERR	ERR	ERR	ERR
-120 F		6	70	14	20.4	59	98
-150 F		3	68	11	15.5	65	74
-175 F		0	ERR	ERR	ERR	ERR	ERR
Water Content	Wt%	13	2.0	0.3	14.4	1.5	3.4
Elemental Analysis	Wt%-Dry						
	C	14	85.8	1.6	1.9	82.6	91.0
	H	14	8.9	0.2	2.7	8.4	9.7
(By Difference)	O	13	4.1	1.5	35.1	0.0	6.6
	N	14	0.6	0.1	20.0	0.4	1.0
	S	13	0.5	0.0	10.7	0.3	0.6
	Sum	13	100.1	0.1	0.1	100.0	100.8
Atomic H/C Ratio		14	1.2	0.0	3.7	1.1	1.4
Heating Value(HHV)	Btu/lb	11	16445.5	- 61.1	0.4	16200.0	16510.0
Gross Component Analysis	Wt%						
BTX/Lt Aromatics		3	4.3	6.0	139.0	1.8	7.6
Phenols		5	1.5	0.3	18.1	1.3	1.9
Cresols		5	3.8	0.6	15.5	3.1	4.4
Methoxyphenols		2	0.5	0.4	99.8	0.4	0.5
Ethylphenol		5	0.9	0.2	19.5	0.7	1.1
Xylenol		5	2.8	0.4	14.2	2.4	3.2
Naphthalene Isomers		2	9.6	50.8	531.5	3.9	15.2
CCR	Wt%	6	3.3	0.4	13.4	2.6	4.0

Table 8.
Comparison of ASTM D-1160 & D-2887 Tar Oil Distillation Data

ASTM D-2887 Tar Oil Distillation Data

		w/ Amoco 6/18/87 data				w/o Amoco 6/18/87 data			
		sample size	AVERAGE (deg F)	95% Confidence Limit		sample size	AVERAGE (deg F)	95% Confidence Limit	
Distillation (ASTM D-86)	LV %			(+/-)	(%)			(+/-)	(%)
	IBP	5	216	18	8.1	4	212	21	9.8
	5	3	320	14	4.3	2	317	13	4.3
	10	5	354	3	1.0	4	353	2	0.6
	20	3	397	4	1.1	3	397	4	1.1
	30	5	433	15	3.4	4	433	21	4.9
	50	5	521	14	2.8	4	523	20	3.8
	70	5	626	22	3.6	4	630	28	4.5
	80	3	693	33	4.7	3	693	33	4.7
	90	5	790	40	5.0	4	799	47	5.9
	95	3	826	22	2.6	2	833	13	1.6
	99	5	948	55	5.7	4	963	59	6.2
	% REC	4	100	1	0.7	3	100	1	1.2
Specific Gravity	60/60 F	5	1.017	0.004	0.4	4	1.018	0.005	0.4
	API	5	7.6	0.5	6.7	4	7.5	0.6	8.2

ASTM D-1160 Tar Oil Distillation Data

		w/ Chromaspec 5/1/85 data				w/o Chromaspec 5/1/85 data			
		sample size	AVERAGE (deg F)	95% Confidence Limit		sample size	AVERAGE (deg F)	95% Confidence Limit	
Distillation (ASTM D-86)	LV %			(+/-)	(%)			(+/-)	(%)
	IBP	4	130	31	23.8	3	126	52	41.5
	5	4	184	22	11.8	3	177	22	12.4
	10	4	227	99	43.6	3	191	23	12.1
	20	2	304	880	289.6	1	206	----	----
	30	4	300	167	55.7	3	240	19	7.8
	50	4	375	155	41.2	3	319	26	8.3
	70	4	483	170	35.2	3	422	36	8.6
	80	2	616	1137	184.7	1	489	----	----
	90	1	595	----	----	1	595	----	----
	95	3	697	216	31.0	2	636	117	18.4
	99	0	n.a.	n.a.	n.a.	0	n.a.	n.a.	n.a.
Specific Gravity	60/60 F	4	1.016	0.008	0.829	3	1.007	0.019	1.905
	API	4	7.8	1.2	14.8	3	9.1	2.7	29.6

APPENDIX A

GPGP Process Flow Diagrams

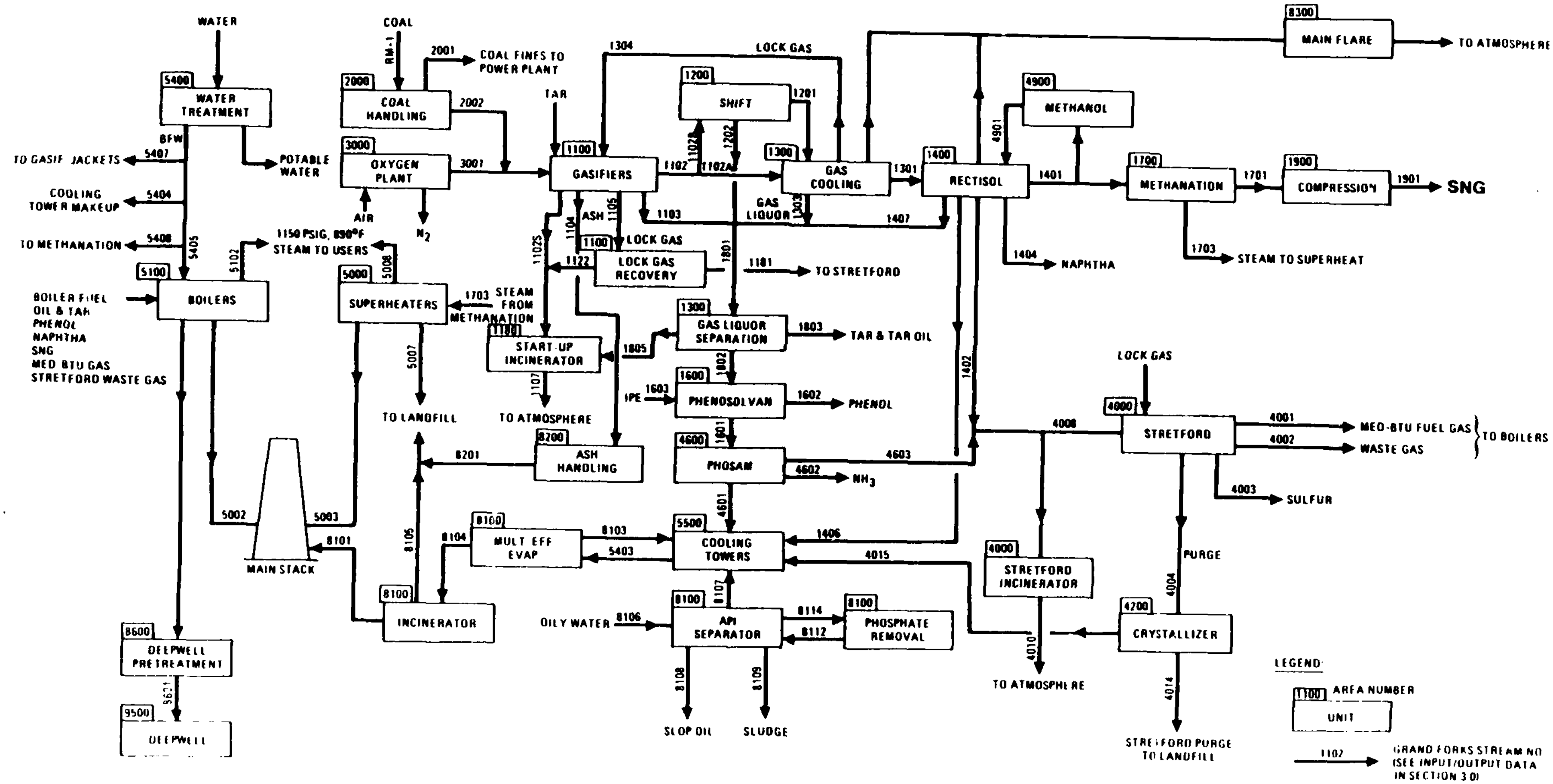


FIGURE A-1. GREAT PLAINS GASIFICATION PLANT PROCESS BLOCK FLOW DIAGRAM.

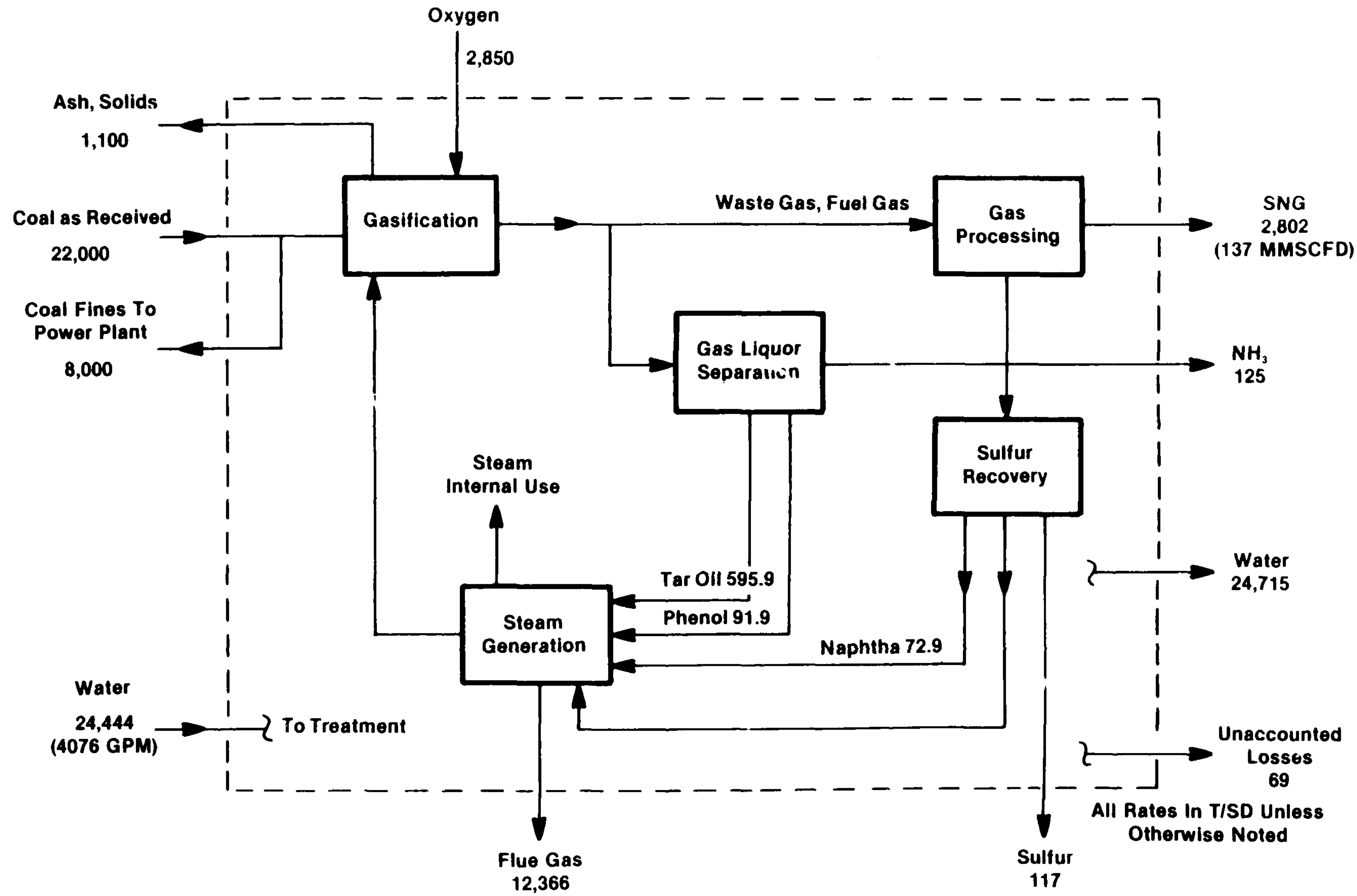


Figure A-2. Overall Process Material Balance @ Design SNG Rate of 137.5 MMSCFD.

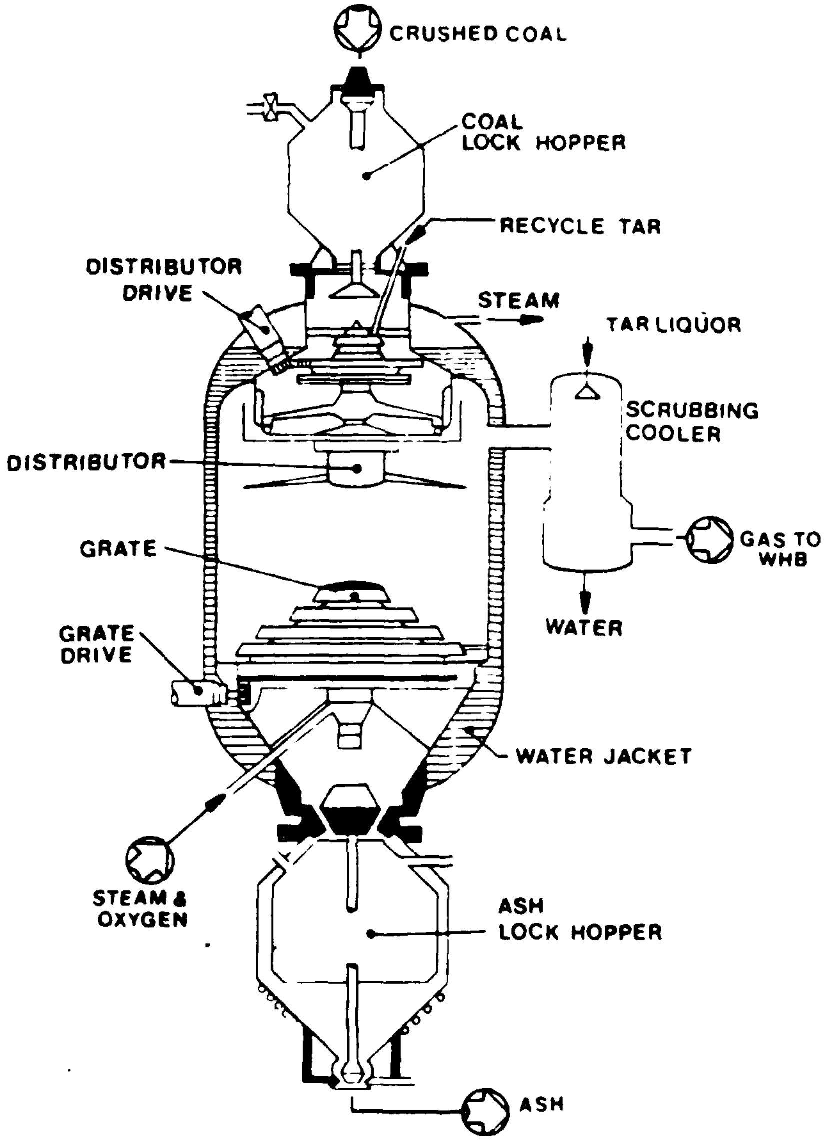


FIGURE A-3. LURGI DRY ASH GASIFIER.

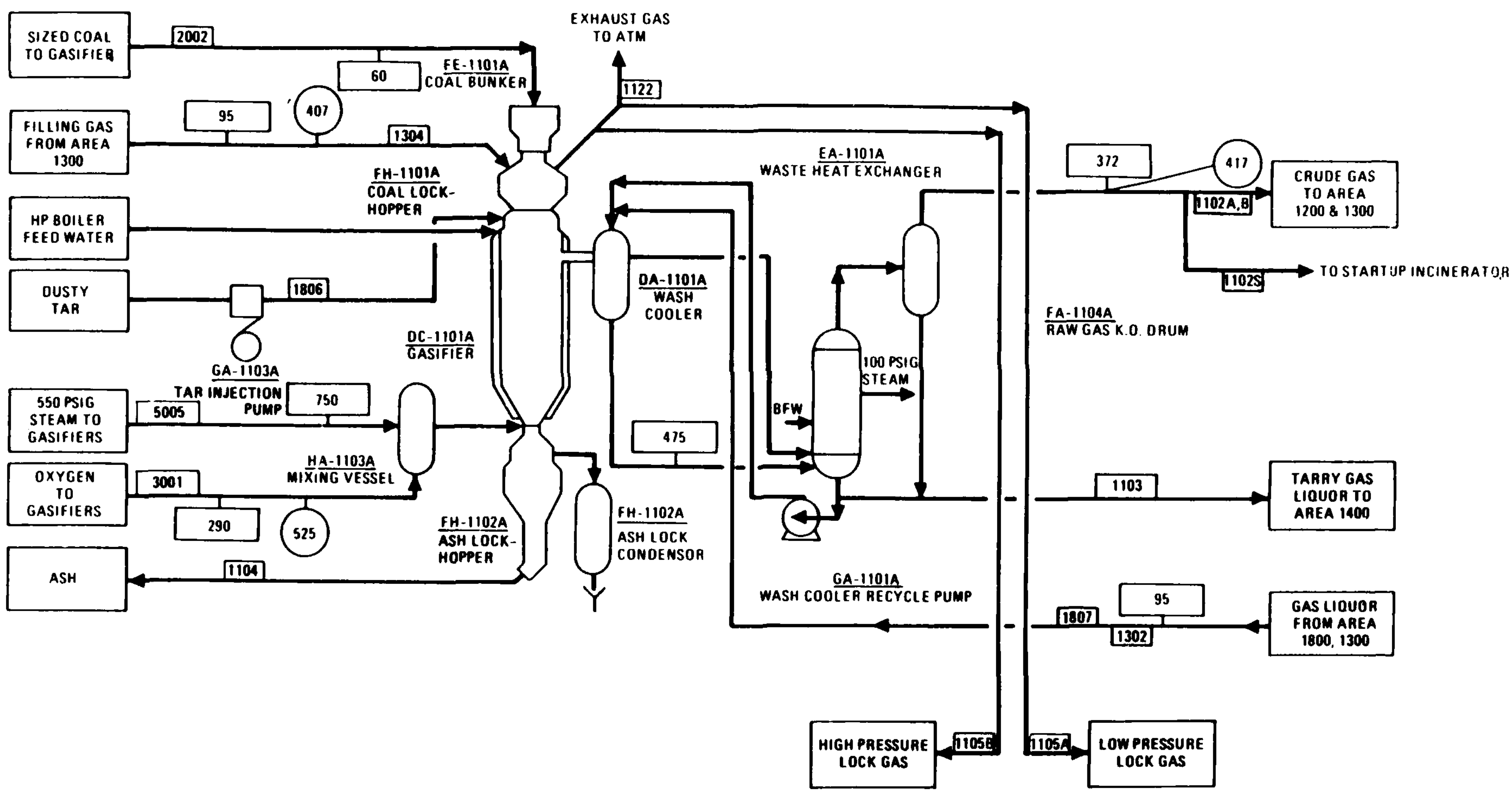


FIGURE A-4. GASIFICATION AND QUENCH - SIMPLIFIED PROCESS FLOW DIAGRAM.

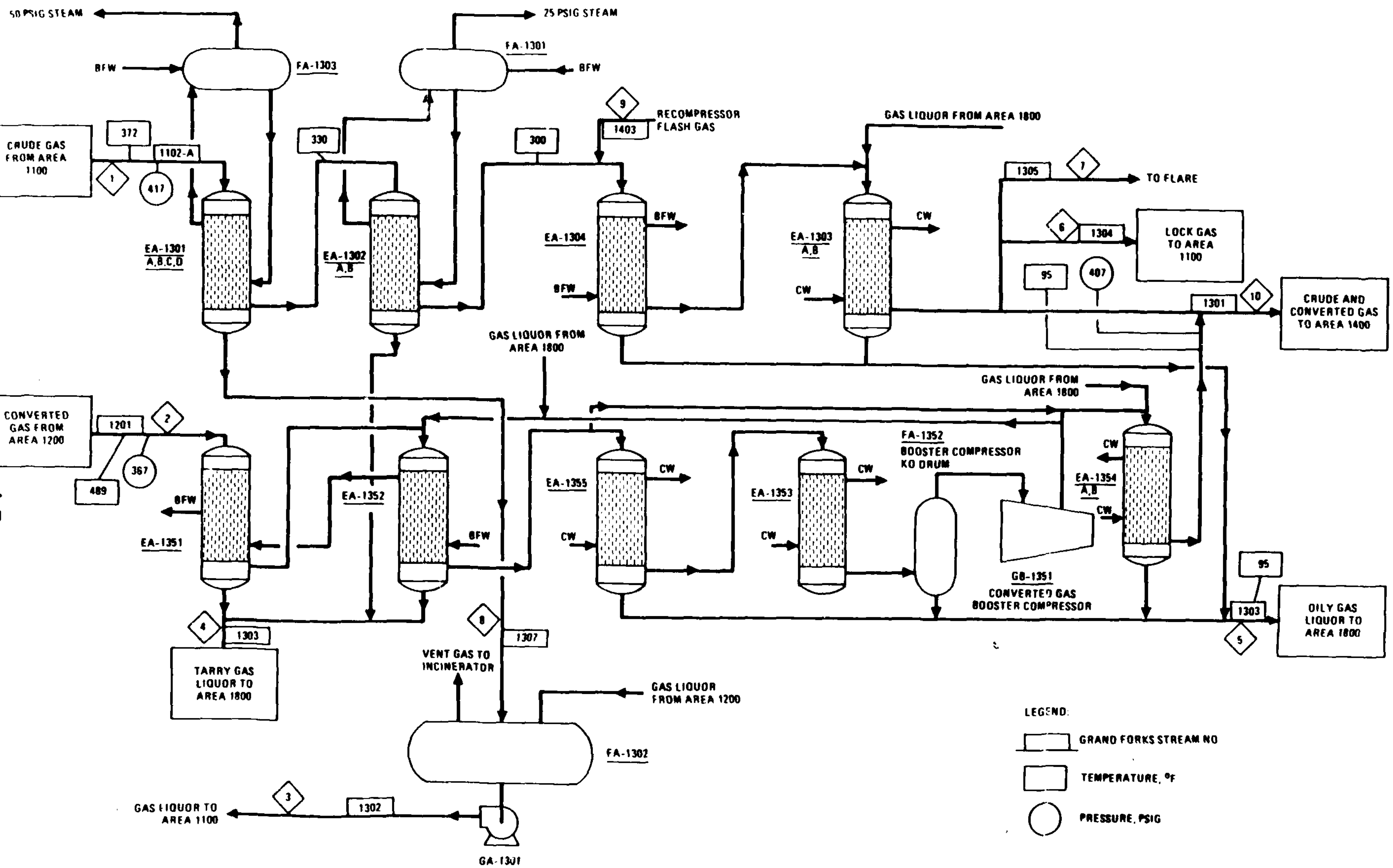


FIGURE A-5. GAS COOLING - SIMPLIFIED PROCESS FLOW DIAGRAM.

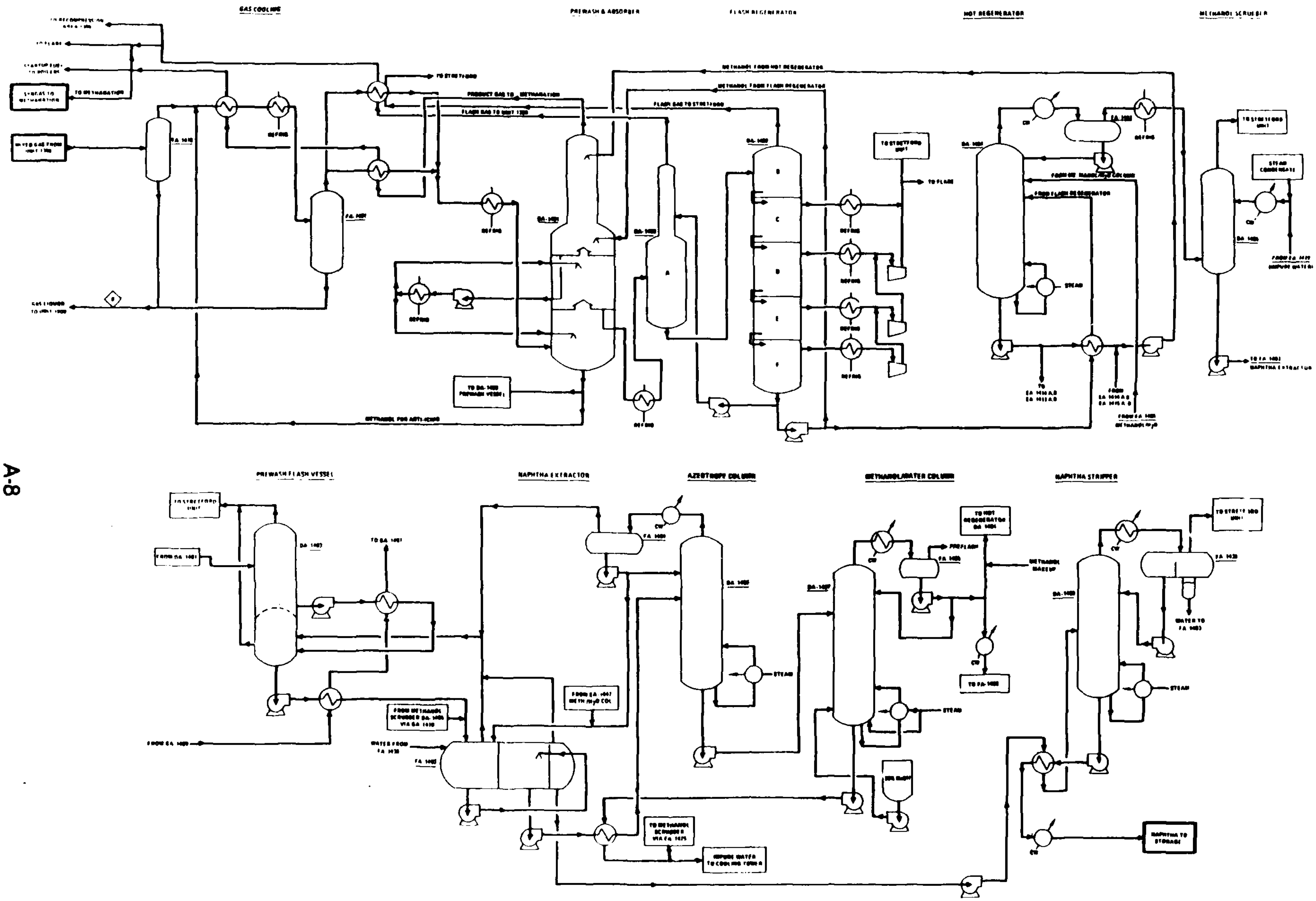


FIGURE A-6. RECTISOL UNIT - SIMPLIFIED PROCESS FLOW DIAGRAM.

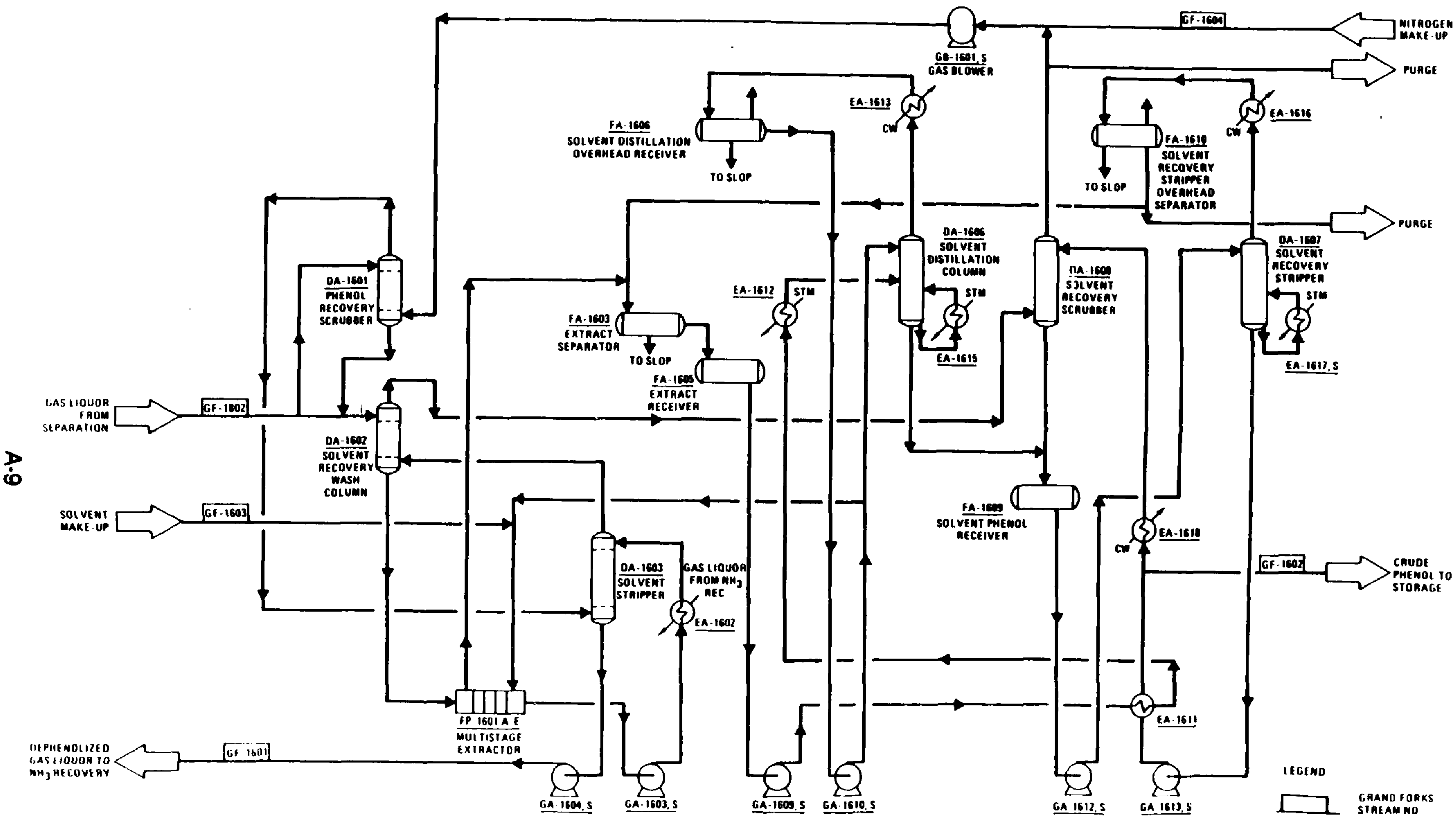


FIGURE A-7. PHENOSOLVAN UNIT SIMPLIFIED PROCESS FLOW DIAGRAM.

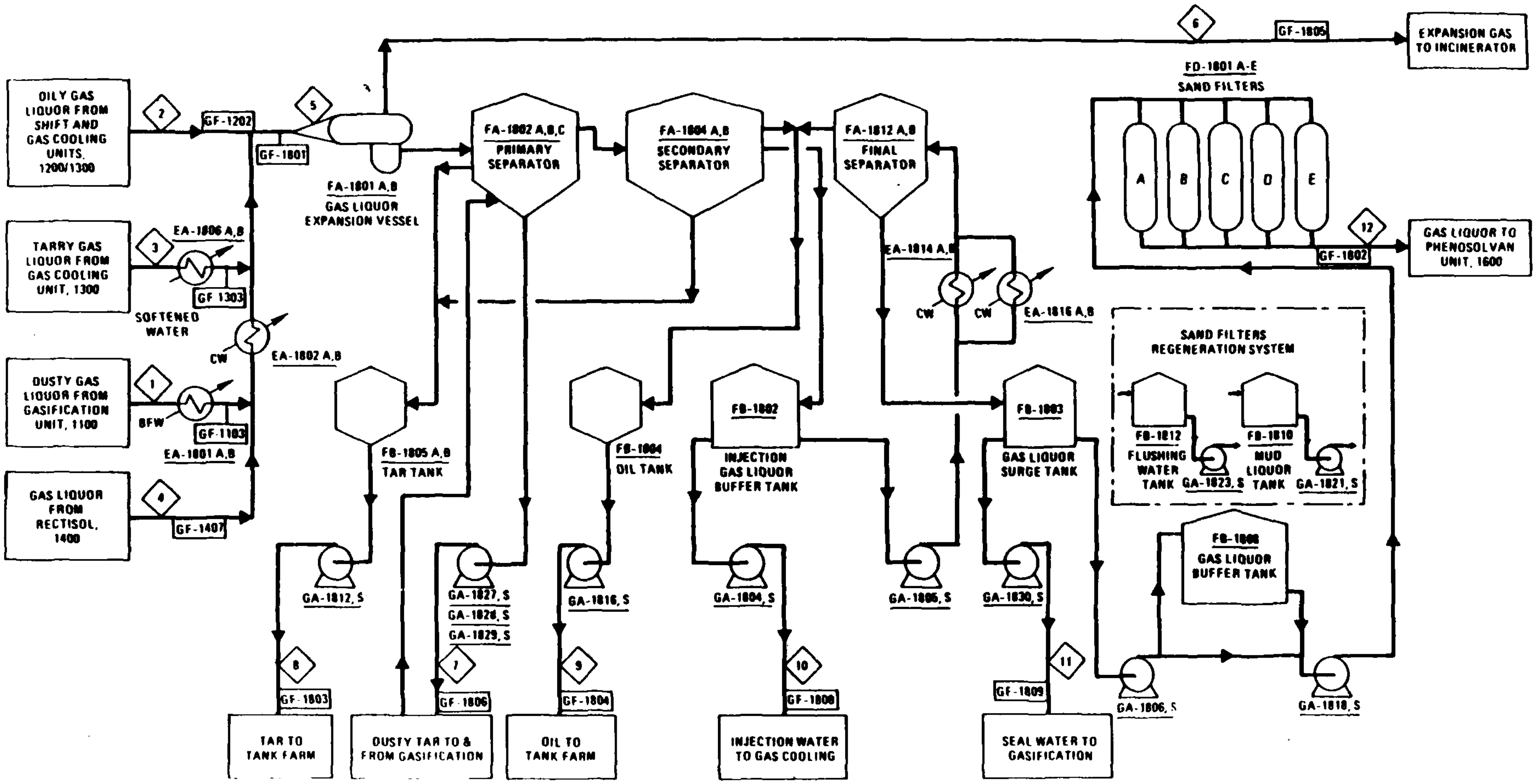


FIGURE A-8. GAS LIQUOR SEPARATION SIMPLIFIED PROCESS FLOW DIAGRAM.

APPENDIX B

Selected Jet Fuel Production Program GPGP Liquid By-Product — Analytical Data

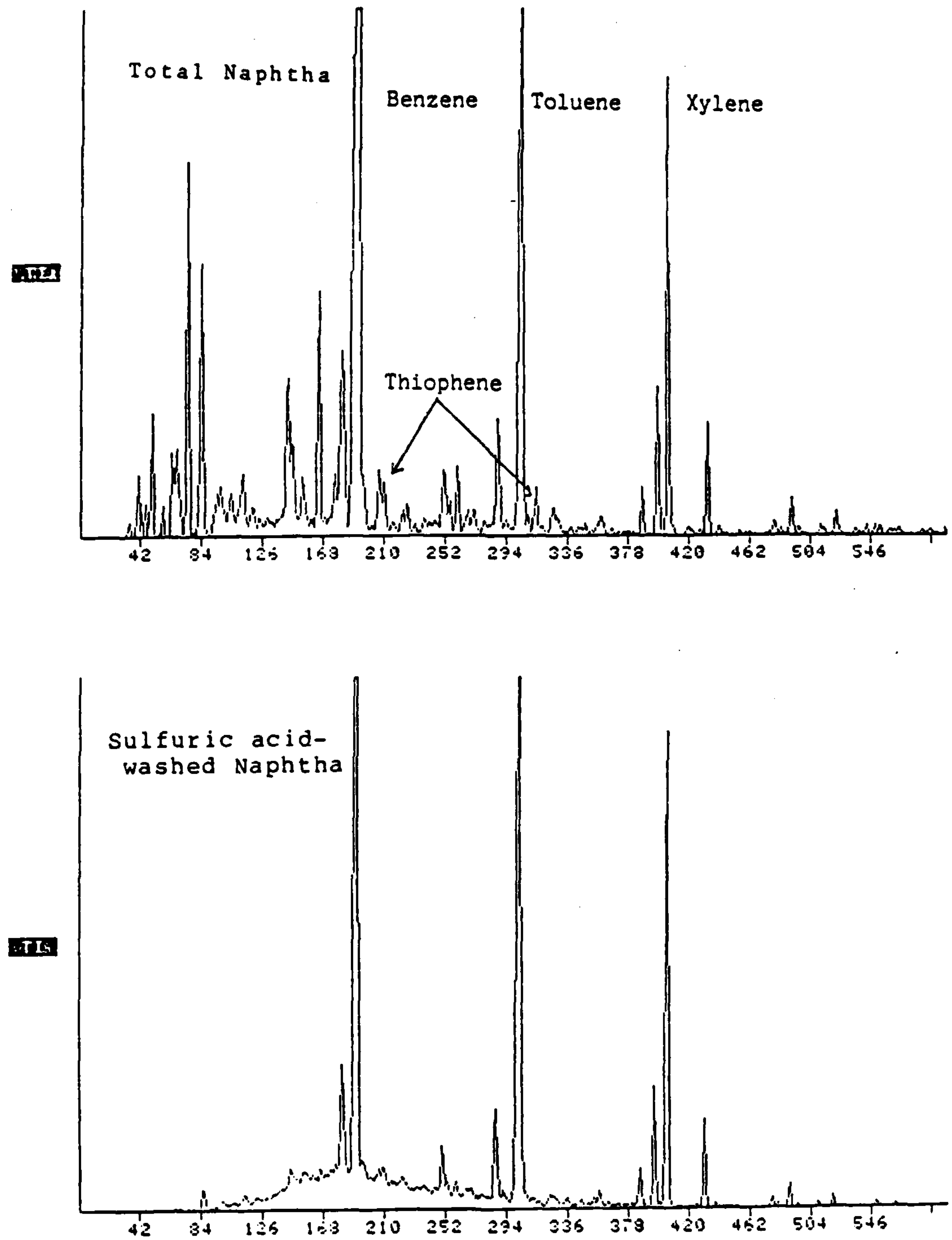
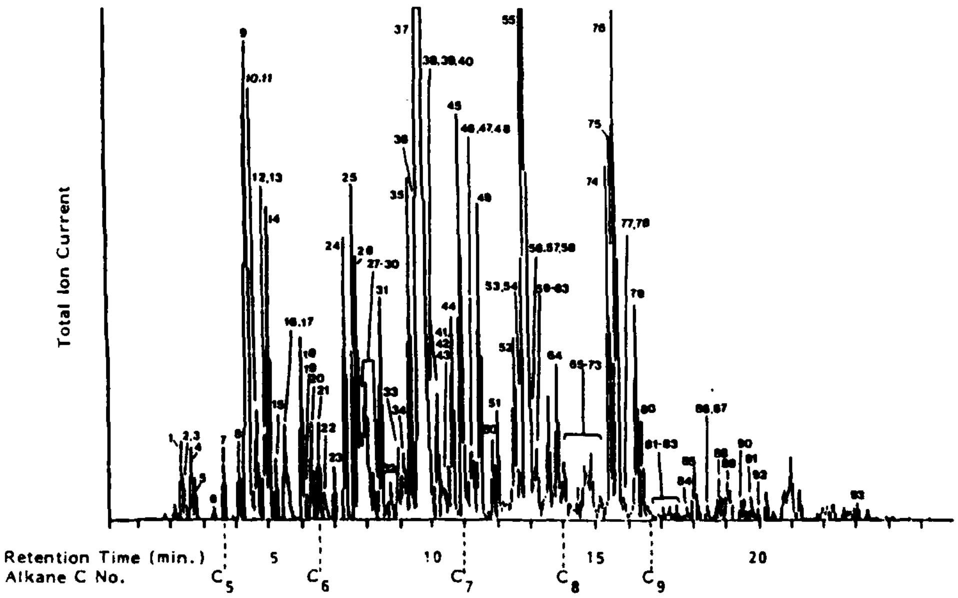
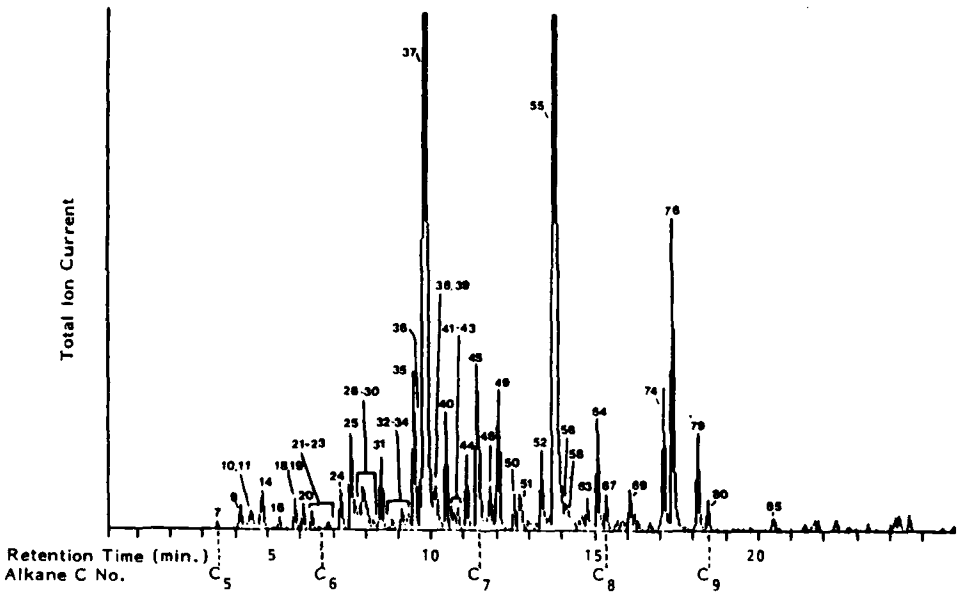


FIGURE B-1. COMPARISON OF GC/MS ANALYSIS OF RAW AND H_2SO_4-WASHED RECTISOL NAPHTHA. (UNDEMRC 2/17/87 - 5/16/87 Quarterly)



GC/MS analysis of rectisol naphtha.



GC/MS analysis of deodorized rectisol napntha.

FIGURE B-2. COMPARISON OF UNDEMRC GC/MS ANALYSIS OF RAW- AND DEODORIZED RECTISOL NAPHTHA.

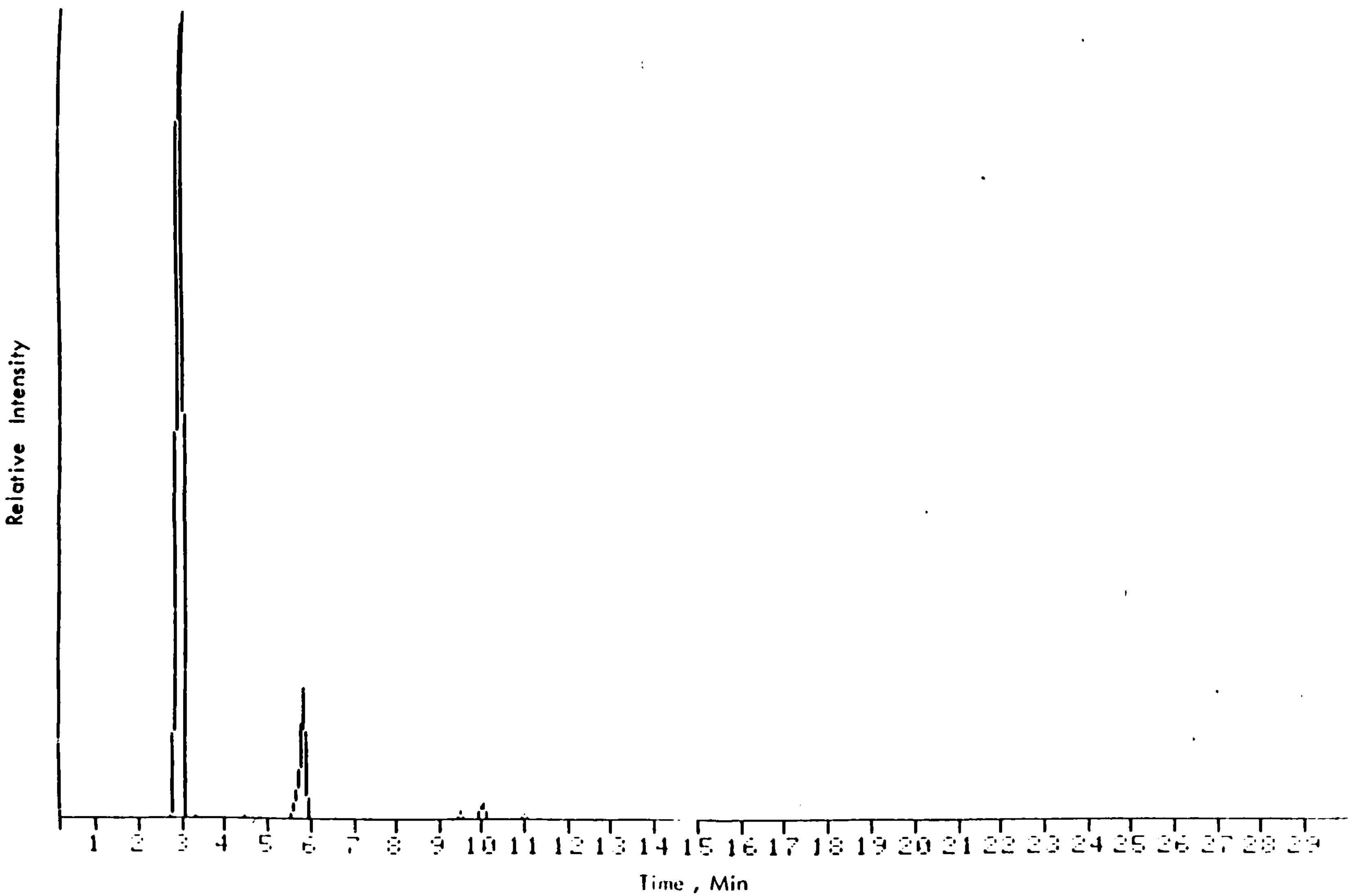


FIGURE B-3. RECONSTRUCTED WRI ION CURRENT CHROMATOGRAM OF THE NAPHTHA STREAM.

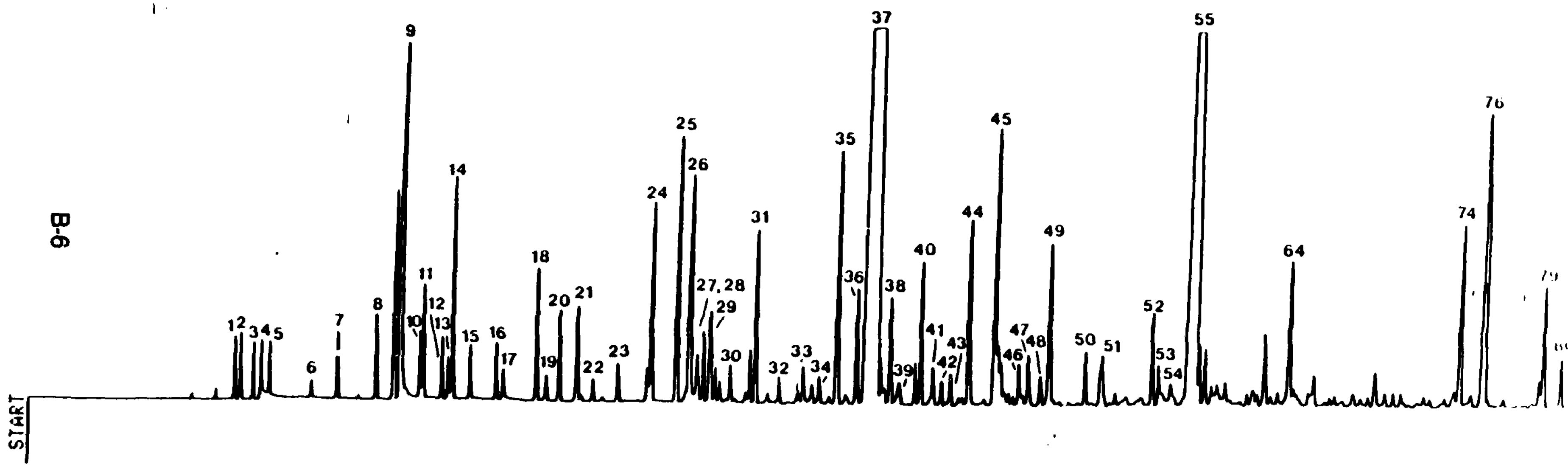


FIGURE B-4. UNDEMRC GC-MS ANALYSIS OF RECTISOL NAPHTHA STREAM.

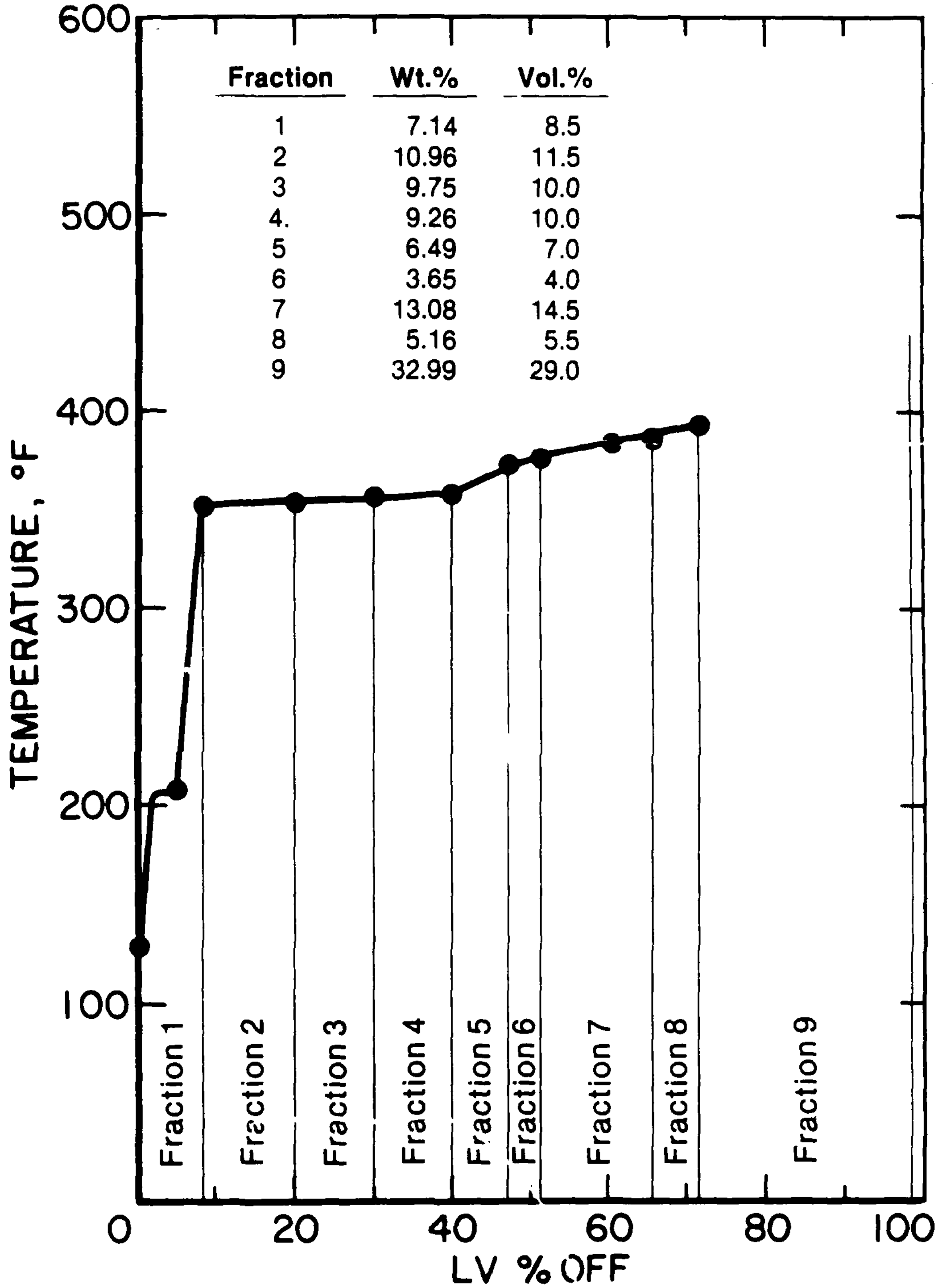


Figure B-5. UNDEMRC Crude Phenol TBP Distillation. (fractions submitted for GC/MS, C_{13} NMR, and Proton-NMR Analysis)

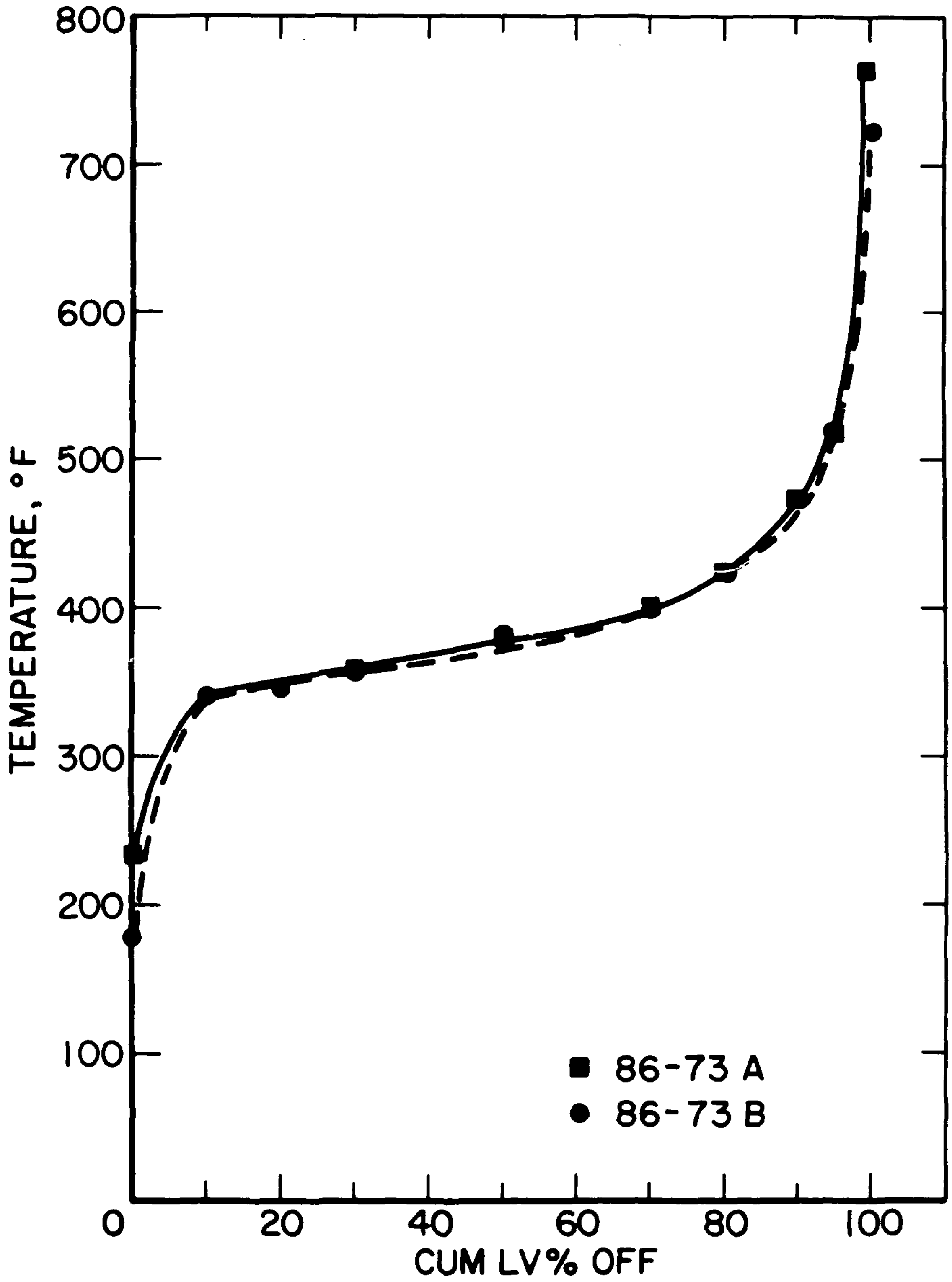


Figure B-6. D-2887 GC Distillation of GPGP Crude Phenol (WRI Samples 86-73A and 86-73B).

NO./16.371

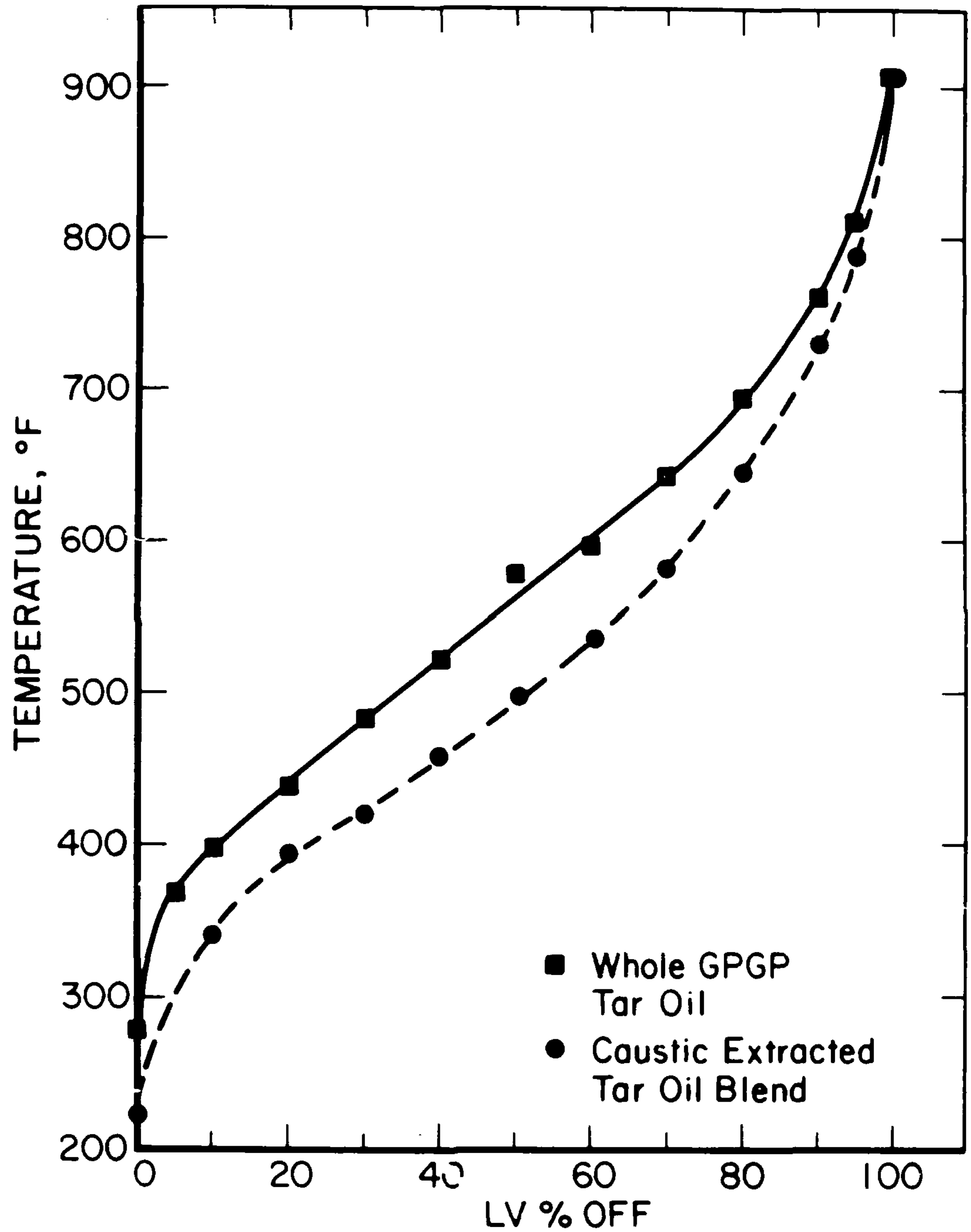


Figure B-7. Comparison of Whole- and Caustic Extraction Tar Oil Blend.

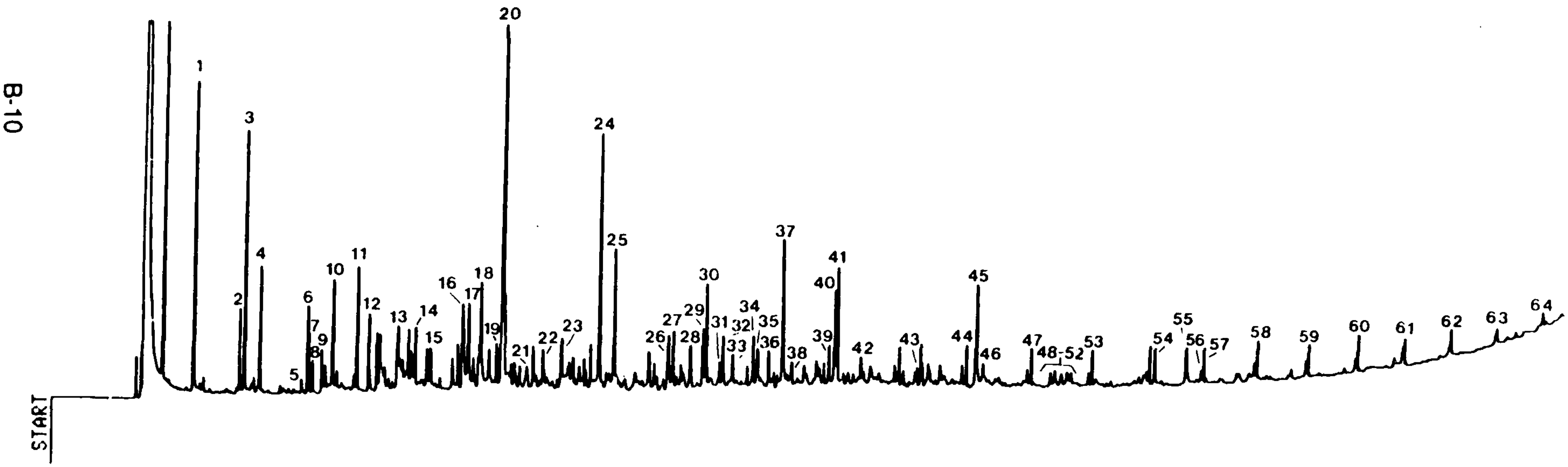


FIGURE B-8. UNDEMRC GC-FID ANALYSIS FOR TAR OIL STREAM.

APPENDIX C

Selected GPGP Jet Fuel Production Program Liquid By-Product Characterization Data

TABLE C-1.
COMPARISON OF AMOCO CAUSTIC — AND CAUSTIC/METHANOL EXTRACTED GPGP NAPHTHA SAMPLES

Sample Date	5/11/87		6/18/87	
Pretreatment	None	NaOH Extracted	None	NaOH/MeOH Extracted
API Gravity	38.9	39.5	40.0	
Elemental Analysis, Wt.%				
Carbon	83.75	86.33	84.11	86.21
Hydrogen	9.94	9.57	9.93	9.92
Nitrogen	0.21	0.06	0.21	0.13
Sulfur	1.73	1.26	1.68	0.86
Oxygen (by difference)	4.37	2.78	4.07	2.88
PONA, Vol.%				
Paraffins				
Normal	—	5.2	6.0	—
ISO	—	2.2	2.4	—
Olegins				
Normal	—	7.3	8.0	—
ISO	—	7.1	7.6	—
Naphthenes	—	13.6	12.1	—
Aromatics	—	64.6	63.9	—
Component Analysis, Wt.%				
Water	0.56	—	0.42	—
Methanol	2.27	—	2.47	—
Acetone	6.32	—	—	—
MEK	3.32	—	—	—
Benzene	45.62	—	46.04	—
Toluene	17.93	—	15.99	—
Xylene	3.25	—	3.60	—
Wt.% C_A (NMR)	65.9	—	—	—
Viscosity @ 25°C, cP	0.46	—	—	—
RVP, psi	*	5.5	—	—
Octane-Research	*	*	—	—
Motor	*	*	—	—
ASTM D-86, °F				
IBP	111	114	—	—
5%	134	158	—	—
10%	142	165	—	—
30%	167	179	—	—
50%	183	188	—	—
70%	*	203	—	—
90%	*	243	—	—
95%	*	267	—	—
FBP	*	341	—	—
% Loss	*	0.1	—	—
% Residue	*	0.2	—	—

*Not done due to odor.

NO./16.387

TABLE C-2.
GPGP RECTISOL NAPHTHA COMPONENT DATA
(UNDEMRC 2/17/87 - 5/16/87 Quarterly)

GC #	b.p. deg.C	------ deg.F	Component	Area% Over	Retention Time	Peak Area%	Elemental, atomic C	H	N	O	S
1				0	25.79	0.26	4	8			
2				0	26.45	0.22	4	8			
3				1	28.08	0.15	4	8			
4	6.0	43	Methanethiol	1	29.04	0.21	1	4			1
5				1	30.10	0.15	4	8			
6				1	35.46	0.00	5	10			
7	36.0	97	n-Pentane	1	38.65	0.24	5	12			
8				2	43.45	0.31	5	10			
9	52.6	127	Acetone	4	46.39	2.28	3	6		1	
10				4	48.27	0.25	5	8			
11				5	49.13	0.44	5	10			
12				5	51.02	0.25	5	10			
13	37.3	99	Thiobismethane	5	52.17	0.16	2	6			1
14				6	52.71	0.86	5	10			
15	37.0	99	Ethanethiol	6	54.93	0.21	2	6			1
16				6	58.26	0.24	5	6			
17				6	59.13	0.15	5	8			
18				7	63.36	0.55	5	8			
19				7	64.61	0.13	6	12			
20				7	66.27	0.42	5	10			
21				8	68.52	0.47	6	12			
22				8	70.51	0.11	6	14			
23				8	73.59	0.18	6	14			
24				9	77.65	0.91	6	12			
25	68.0	154	n-Hexane	10	80.79	1.24	6	14			
26	79.6	175	2-Butanone	12	82.37	1.45	4	8		1	
27				12	83.22	0.33	6	12			
28				13	84.00	0.33	6	10			
29				13	84.82	0.61	6	12			
30				13	86.00	0.11	6	12			
31				14	90.49	0.81	6	12			
32				14	93.42	0.00	6	12			
33				14	96.44	0.21	6	8			
34				14	98.45	0.17	6	8			
35				16	100.69	1.30	6	10			
36				16	103.07	0.60	6	12			
37	80.1	176	Benzene	63	105.60	46.60	6	6			
38	84.2	184	Thiophene	63	107.13	0.57	4	4			1
39				64	108.27	0.18	6	8			
40	83.0	181	Cyclohexene	64	110.85	0.66	6	10			
41				65	112.31	0.20	7	14			
42				65	113.38	0.13	7	14			

TABLE C-2.
GPGP RECTISOL NAPHTHA COMPONENT DATA (CONTINUED)

GC #	b.p. deg.C	------ deg.F	Component	Area% Over	Retention Time	Peak Area%	Elemental, atomic C	H	N	O	S
43				65	114.47	0.15	7	14			
44				66	116.70	1.05	7	14			
45				67	119.97	1.51	7	12			
46				68	122.95	0.24	7	14			
47				68	124.13	0.30	7	12			
48				68	125.75	0.21	7	14			
49				69	126.82	0.98	7	14			
50	98.4	209	n-Heptane	69	131.44	0.27	7	14			
51				70	133.58	0.44	7	12			
52				70	139.89	0.47	7	12			
53				70	140.62	0.21	7	12			
54				71	141.05	0.13	8	16			
55	110.6	231	Toluene	88	145.17	17.57	7	8			
56				88							
57				88							
58				88							
59			#56 - 63 total	90		1.61					
60				90							
61				90							
62				90							
63				90							
64	126.0	259	C8H18	91	156.81	0.83	8	18			
65				91							
66				91							
67				91							
68				91							
69			#65 - 73 total	91		0.79					
70				91							
71				91							
72				91							
73				91							
74			C2 Benzene	92	177.95	0.94	8	10			
75				92		0.01					
76			C2 Benzene	95	180.85	2.50	8	10			
77			#77 - 78 total	95		0.18					
78				95							
79			Benzene-C2	96	188.57	0.62	8	10			
80	150.8	303	C9H20	96	190.83	0.27	9	20			
81				96							

TABLE C-3.
Proton and Carbon-13 NMR Analyses of GPGP Rectisol Naphtha (UNDEMRC 2/17/87 - 5/16/87 Quarterly)

Proton NMR

Carbon Type	% Area
Aromatic	38.9
Phenol	0.4
Acenapthene	1.9
$-CH_2$-alpha	22.2
$-CH_2$-beta	10.5
$-CH_2-$	13.9
$-CH_3$	12.2
	100.0

Carbon-13 NMR

Carbon Type	% Area
Aliphatic, C=	2.0
Aromatic, C=O	1.1
Phenolic	0.0
Aromatic, =C=	2.7
Aromatic, =C-	66.7
Methoxy	1.1
Aliphatic, $-CH_2-$	4.3
+C	9.7
alpha C	8.0
$-CH_3$	4.4
	100.0

TABLE C-4.
ANALYSIS OF C_5^- LOSSES FROM GPGP RECTISOL NAPHTHA
(5/11/87 samples from Amoco 8/26/87 presentation)

		Can Sample	"Bomb" Sample
Wt%	C_1	0	0.001
	C_2	0	0
	C_3	0.0006	0.058
	$C_3^=$	0	0
	iC_4	0.014	0.038
	nC_4	0.081	0.187
	$C_4^=$	0.438	1.064
	iC_5	0.158	0.196
	nC_5	0.469	0.603
	Cyclo-C_5	0.241	0.280
	$C_5^=$	2.258	2.735
	$C_5^{==}$	3.507	4.099

TABLE C-5.
Results From WRI Analysis of GPGP Rectisol Naphtha Sample REF 86-74[1]

Specific Gravity, 60/60°F	0.821
Elemental Composition, wt% (ppm)	
Carbon	85.0
Hydrogen	9.8
Nitrogen	0.18 (2071)
Sulfur	1.8 (18270)
Oxygen (difference)	3.2
Distillation (ASTM-D2887)	
IBP	62°F
5/10%	96/126
20/30	161/169
40/50	173/177
60/70	187/221
80/90	228/242
95	276
FBP	364

Benzene/Alkylbenzenes by GC/MS

Carbon Number	% of Total Ionization
6	73.6
7	14.7
8	2.5
9	0.6

[1]From October 1987 WRI Quarterly Technical Progress Report.

TABLE C-6.
COMPARISON OF GPGP CRUDE PHENOL GC COMPONENT ANALYSES BY HRI ,ANG, AND OUTSIDE LABORATORY

	Outside Lab	HRI
pyridines (total)	1.1	
neutral oil	3.2	
catechol	11.3[1]	
residue (>180°C @ 38 mm Hg)	21.7[2]	5.3*
lights	1.9	1.9
phenol	31.0	45.1
guaiacol	0.8	2.7
ortho-cresol	6.1	8.7
2,6-xylenol	0.4	0.5
para-cresol	6.3	9.2
meta-cresol	8.9	13.7
ortho-ethyl phenol	0.4	0.9[3]
2,4-xylenol	1.1	2.1
2,5-xylenol	0.8	
para-ethyl phenol	1.1	1.8
meta-ethyl phenol	0.8	1.5
2,3-xylenol	0.4	2.7
3,5-xylenol	1.1	
3,4-xylenol	0.5	3.9
unknowns	1.1	
closure	100.0	100.0

*chromatographic method

[1]From lead precipitation test, may include hydroquinones and resorcinol.

[2]Based on Rotovap distillation

[3]Includes Hydroquinone

TABLE C-7.
ELEMENTAL ANALYSIS OF GPGP CRUDE PHENOL D-86 FRACTIONS (UNDEMRC 2/17/87 - 5/16/87 Quarterly)

SPECIFIC GRAVITY, 1.075:

VOL.%	TEMPERATURE deg.F	deg.C	WT% OF FRAC.	WT% C	WT% H	WT% N	WT% O+S (diff)	SUM
IBP	207	97						
5%	374	190	4.88	73.01	7.47	0.20	19.32	100
10%	374	190	4.87	76.22	7.19	0.00	16.59	100
20%	381	194	9.73	76.78	7.13	0.00	16.09	100
30%	383	195	9.66	76.29	7.10	0.10	16.51	100
40%	387	197	9.64	76.23	7.16	0.17	16.44	100
50%	394	201	9.74	76.57	7.28	0.16	15.99	100
60%	410	210	9.51	77.33	7.38	0.13	15.16	100
70%	433	223	9.73	77.14	7.46	0.19	15.21	100
80%	468	242	9.64	75.31	7.55	0.28	16.86	100
90%	496	258	10.91	73.72	7.55	0.41	18.32	100
94.2%	496	258	4.22	73.22	7.60	0.72	18.46	100
RESIDUE			6.23	83.78	5.73	1.59	8.90	100
TOTAL WT%			98.76	75.39	7.15	0.28	15.93	
ORIGINAL SAMPLE WT%			100.0	76.03	7.34	0.31	16.32	
RECOVERY %			98.8	99.2	97.4	90.3	97.6	

TABLE C-8.
PROTON NMR DATA FOR TOTAL CRUDE PHENOL AND THE SUM OF THE FRACTIONS

FUNCTIONAL GROUP	NMR REGION ppm	TOTAL PROTON Area %	SUM PROTON Area %
Aromatic	9.0-5.9	54.8	50.1
Phenolic	5.2-4.4	12.2	12.0
Methoxy	4.4-3.5	1.3	2.0
Alpha-Meth	3.3-1-9	23.6	24.2
Other		8.0	10.3
Total		99.9	98.6

	Fraction 1	Fraction 2	Fraction 3	Fraction 4	Fraction 5	Fraction 6	Fraction 7	Fraction 8	Fraction 9
Temp., °C	IBP-178	178-179	179-180	180-181	181-190	190-193	193-202	198-202	Pot Residue
wt.% of total	7.14	10.96	9.75	9.26	6.50	3.65	13.08	5.16	33.00
NMR, area %									
Aromatic	30.1	77.3	75.1	72.7	62.8	52.6	47.1	44.0	33.2
Phenolic	4.0	15.4	15.2	15.3	13.5	11.0	10.9	10.5	11.8
Methoxly	5.8	0.9	0.6	1.0	1.5	2.0	3.1	3.7	1.6
Alpha-Meth	21.7	3.9	6,4	8.2	19.0	31.0	35.1	37.1	36.2
Other	38.4	2.5	2.7	2.8	3.2	3.4	3.8	4.7	17.2
Total	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0	100.0

TABLE C-9. UNDEMRC PROTON NMR ANALYSIS OF CRUDE PHENOL TBP FRACTIONS

Compound		#[b]	1	2	3	4	5	6	7	8	9
Type	RT[a]	wt%[c]	7.1	11.0	9.8	9.3	6.5	3.6	13.1	5.2	33.0
Aniline	12.70							.49	.48		
Phenol	12.79		47.62	95.38	87.72	84.81	58.20	22.73	5.82	1.01	
C1 Phenol	14.97		12.96	4.10	10.58	13.00	24.30	20.08	9.04	1.22	.06
C1 Phenol	15.73		2.60	.381	1.26	1.62	14.53	49.08	71.38	65.44	3.54
Guaiacol	16.08		4.19		.02	.03	.43	2.04	4.17	6.13	.57
C2 Guaiacol	16.51		1.76	.086	.06	.13	.62	1.05	.89	.41	
C2 Phenol	17.31		.65	.025	.02	.01	.39	1.31	2.07	2.36	.22
C2 Phenol	17.61		.81		.03	.03	.16	.56	2.15	9.21	2.48
C2 Phenol	17.64							7.48	2.09	5.06	2.12
C2 Phenol	18.12								.13	2.46	6.60
C2 Phenol	18.19								.31	2.71	12.26
C2 Phenol	18.43							.02	.16	1.16	1.76
C3 Phenol	18.64								.06	.19	.06
Naphthalene	18.70		1.34	.03	.08	.07	.24	.51	.60	.62	.38
C2Phenol	18.23										4.39
	18.87		.37							.22	2.04
Resorcinol	18.99									.07	2.32
C3 Phenol	19.14									.10	
C3 Phenol	19.59									.04	2.13
C3 Phenol	19.75									.08	1.80
C1-diol	21.38										6.84
C1-diol	22.64										1.79
C2-diol	22.83										1.31
C2-diol	23.67										2.09
	23.78										2.48
Naphthol	26.85										3.26

[a] Retention time.
[b] Fraction number.
[c] The wt% of total sample.

TABLE C-10. UNDEMRC GC/MS COMPONENT ANALYSIS OF GPGP CRUDE PHENOL TBP FRACTIONS

AD-A198 700 PRODUCTION OF JET FUELS FROM COAL DERIVED LIQUIDS VOLUME 4 GPGP JET FUELS..(U) BURNS AND ROE SERVICES CORP PITTSBURGH PA R J ROSSI JUL 88 2/2

UNCLASSIFIED AFWAL-TR-87-2042-VOL-4 FY1455-86-N0657 F/G 21/4 NL

END
DATE
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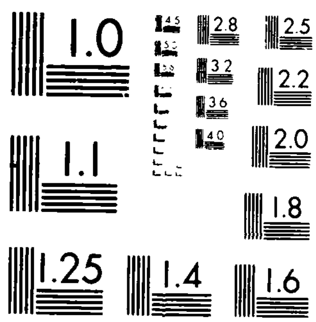

MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS 1963-A

TABLE C-11.
RESULTS FROM WRI FLASH DISTILLATION OF GPGP CRUDE PHENOL

		Crude Phenol (Ref. 86-73A)	Flash Distillation Product IBP-450°F	Flash Distillation Product 450°F +
Recovery	Vol.%	(Feed)	81.0	12.3
Specific Gravity	60/60°F	1.066	1.063	1.099
Water Content	Wt.%	5.5	N.R.	N.R.
Elemental Composition	Wt.%			
Carbon		71.30	70.20	73.40
Hydrogen		7.40	7.30	7.10
Oxygen (by Difference)		20.69	21.96	18.29
Nitrogen		0.47	0.34	0.79
Sulfur		0.14	0.20	0.42
Distillation (ASTM D-2887) °F				
IBP		233	167	323
5%		—	335	330
10		341	340	333
20		340	346	360
30		358	350	371
40		—	356	400
50		382	372	436
60		—	382	463
70		401	389	492
80		424	407	540
90		476	448	660
95		518	481	747
FBP		766	613	892

TABLE C-12.
SIMULATED GC DISTILLATION OF GPGP TAR OIL[1]

Initial column temp: 60 deg.C — Sample density:
Heating Rate: 6 deg./min to 320 deg.C — 12.2 mg/ml

Carbon Number	Boiling Pt.Range,deg.C	deg.F	Cumulative Area%	Area%	Std. Dev.
7	0 - 98		0.00%	0.00%	
8	98 - 126		2.13%	2.13%	0.42
9	126 - 151		6.34%	4.21%	0.77
10	151 - 174		12.17%	5.83%	0.84
11	174 - 196		23.81%	11.64%	1.64
12	196 - 216		43.22%	19.41%	2.08
13	216 - 235		50.56%	7.34%	0.83
14	235 - 253		59.52%	8.96%	0.81
15	253 - 271		66.17%	6.65%	1.42
16	271 - 287		73.91%	7.74%	0.73
17	287 - 302		79.16%	5.25%	0.92
18	302 - 317		85.67%	6.51%	1.20
19	317 - 331		87.32%	1.65%	0.20
20	331 - 344		90.55%	3.23%	0.24
21	344 - 356		92.31%	1.76%	0.18
22	356 - 369		94.19%	1.88%	0.38
23	369 - 380		95.86%	1.67%	0.24
24	380 - 391		96.42%	0.56%	0.14
25	391 - 402		97.16%	0.74%	0.16
26	402+		99.88%	2.72%	0.60

[1]From May 6, 1987 UNDEMRC Presentation

TABLE C-13.
UNIVERSITY OF UTAH GPGP TAR OIL ANALYSIS

Fraction		Light Fraction <200 C (392 F)	Heavy Fraction >200C (392 F)
% Recovery		47 %	53 %
Elemental Composition	wt%		
C		82.20	84.61
H		8.72	7.92
O (by Difference)		8.15	6.56
N		0.47	0.62
S		0.46	0.29
Pour Point	F	-31	93
Density @ 77 F	g/cc	0.963	1.007
Viscosity @ 77 F	cP	3.75	1300
Metals	wt%		
Zn		0.1	0.6
Cd		0.1	0.3
Ba		0.2	1.4
B		0.2	0.9
Mg		<0.1	4.0
Si		<0.5	15.5
Fe		<0.1	11.0
Ni		<0.1	<0.1
V		<0.1	0.1
TGA Distillation	%		
	10	145	292
	20	174	342
	30	190	370
	40	208	391
	50	217	430
	60	230	460
	70	243	513
	80	257	567
	90	270	666
	100	392	946 (95 % end point)
GC Distillation	% at		
-Gasoline	<200 C	19.[illegible]	n.a.
-Kerosine	200-275 C	58.5	n.a.
-Gas oil	275-325 C	17.9	n.a.
-Heavy gas oil	325-400 C	3.4	n.a.
-Vac gas oil	>400 C	0.6	n.a.
Compound type	wt %		
Aliphatic & Aromatic		38.5	13.0

TABLE C-14.
AMOCO ANALYSIS OF GPGP WHOLE TAR OIL AND FRACTIONS[1]

Sample Date	5/11/87		5/28/87		6/18/87	
Fraction	Total	750-°F	Total	550-°F	Total	
API Gravity	6.6	11.5	6.6	15.4	8.0	
Elemental Analysis, Wt%						
Carbon	82.12	83.63	--	83.46	75.15	
Hydrogen	8.57	8.80	--	8.88	8.73	
Nitrogen	0.87	0.73	--	0.57	0.49	
Sulfur	0.40	0.38	--	0.43	0.36	
Oxygen	8.04	6.46	--	6.66	15.27	
Water, Wt%	2.04	0	--	0	11.38	
Wt% C_A (NMR)	61.4	63.4	--	--	--	
Viscosity @ 25°C, cP	185.10	--	--	--	--	
Pour Point, °F	70	--	--	--	--	
Refractive Index (70°C)	1.5489	--	--	--	--	
Ash Oxide, Wt%	0.03	0.	--	--	0.08	
Ramscarbon, Wt%	2.26	0.30	--	--	1.25	
Solids by Filtration, wt%	0.25	--	--	--	0.60	
PSD: <4.7 Microns	17.6				18.3	
4.7-6.6	17.6				12.7	
6.6-9.4	21.2				15.6	
9.4-13	16.8				10.3	
13-19	17.4				12.0	
19-27	8.4				19.1	
27-38	0.8				12.0	
Distillation, °F						
Type	10mm D-1160	--	--	D-86	10mm D-1160	D-2887
IBP	142			169	139	232
5%	180			240	186	328
10%	196			270	199	359
30%	245			384	245	434
50%	334			410	315	516
70%	441			441	406	609
90%	--			493	623	755
95%	--			549	--	814
FBP	649			563	623	891
% Loss	4.2%			1%	4.8%	--
% Residue	4.8%			1%	3.9%	--
Extraction, wt%						
Oils	97.3	--	--	--	--	
Asphaltenes	1.7	--	--	--	--	
Preasphaltenes	0.7	--	--	--	--	
THF Insolubles	0.3	--	--	--	--	

(1)

TABLE C-15.
ANALYSIS OF FRACTIONS FROM WRI FLASH DISTILLATION AND CAUSTIC EXTRACTION OF GPGP TAR OIL[1]

	REF 86-72A Tar Oil	87-08-3 IBP-450°F	87-08-4 450-750°F	87-08-9 XTR Blend[2]
Specific Gravity, 60/60°F	1.018	0.953	1.028	0.982
Carbon	83.5	80.6	81.7	84.2
Hydrogen	8.6	9.2	8.3	9.0
Nitrogen	0.8	0.6	0.7	0.7
Sulfur	0.5	0.5	0.6	0.6
Oxygen	6.6	9.1	8.7	5.5
Distillation (ASTM D-2887)				
IBP	200	171	278	223
5/10	318/354	235/284	368/397	294/342
20/30	396/423	337/364	438/483	395/422
40/50	469/513	384/401	522/577	459/497
60/70	559/617	412/432	597/644	534/583
80/90	686/774	458/506	695/762	647/731
95	834	543	813	791
FBP	930	674	908	908
Vol % of Tar Oil	100	27	55	74

(1)From October 1987 WRI Quarterly Technical Progress Report

(2)Product of blending caustic-extracted IBP-450°F cut with ~2X its volume of 450-750°F heavy distillate.

TABLE C-16.
ELEMENTAL ANALYSIS OF GPGP TAR OIL D-86 FRACTIONS
(UNDEMRC 2/17/87 - 5/16/87 Quarterly)

SPECIFIC GRAVITY, 1.025:

VOL.%	TEMPERATURE deg.F	deg.C	WT% OF FRAC	WT% C	WT% H	WT% N	WT% O+S (diff)	SUM
IBP	180	82						
5%	275	135	4.49	86.69	10.33	0.56	2.42	100
10%	347	175	4.59	81.62	9.66	0.87	7.85	100
20%	381	194	9.07	80.38	9.59	0.78	9.25	100
30%	405	207	9.37	79.63	9.19	0.67	10.51	100
40%	430	221	9.56	80.88	8.76	0.81	9.55	100
50%	473	245	9.46	83.04	8.86	0.87	7.23	100
60%	511	266	9.27	83.74	8.96	0.85	6.45	100
70%	511	266	9.95	84.64	8.78	0.63	5.95	100
80%	541	283	9.76	85.33	8.91	0.67	5.09	100
90%	565 max	296	10.44	85.32	9.13	0.65	4.90	100
RESIDUE			10.54	90.11	4.90	1.15	3.84	100
TOTAL WT%			96.50	80.90	8.36	0.75	6.49	
ORIGINAL SAMPLE WT%			100.0	82.60	8.63	1.01	7.76	
RECOVERY %			96.5	97.9	96.9	74.4	83.6	

TABLE C-17.
UNDEMRC GC/MS GPGP TAR OIL COMPONENT ANALYSIS

GC #	b.p. deg.C	------ deg.F	Component	Area% Over	Retention Time	Peak Area%	Elemental, atomic C	H	N	O	S
1	111	231	Toluene	8	3.22	8.34	7	8			
2	138	281	C2 Benzene	9	5.40	0.70	8	10			
3	139	282	C2 Benzene	12	5.52	2.56	8	10			
4	144	292	C2 Benzene	13	5.96	1.39	8	10			
5			C3 Benzene	13	7.16	0.18	9	12			
6			C3 Benzene	14	7.33	0.86	9	12			
7	182	359	Phenol	15	7.36	0.66	6	6		1	
8			C3 Benzene	15	7.48	0.32	9	12			
9			C3 Benzene	16	7.75	0.62	9	12			
10			C4 Benzene	17	8.06	1.44	10	14			
11	191	376	C1 Phenol	18	8.76	1.30	7	8		1	
12	202	396	C1 Phenol	19	9.10	1.10	7	8		1	
13			C2 Phenol	21	9.94	1.65	8	10		1	
14			C1 Benzofuran	22	10.78	0.76	9	8		1	
15			C2 Phenol	22	10.88	0.55	8	10		1	
16			C2 Phenol	24	11.77	1.09	8	10		1	
17			C2 Phenol	25	11.94	1.76	8	10		1	
18			C2 Phenol	28	12.28	2.29	8	10		1	
19			C2 Phenol	28	12.54	0.85	8	10		1	
20	218	424	Naphthalene	37	12.88	8.54	10	8			
21			C2 Benzofuran	37	13.38	0.37	10	10		1	
22			C3 Phenol	38	13.75	0.79	9	12		1	
23			C3 Phenol	39	14.56	1.22	9	12		1	
24	240	464	2-Methylnapth.	43	15.62	3.59	11	10			
25	241	466	1-Methylnapth.	45	16.06	1.84	11	10			
26	256	493	Biphenyl	46	17.08	0.90	12	10			
27	254	489	C14H30	47	17.63	1.37	14	30			
28			C2 Naphthalene	48	18.26	0.96	12	12			
29			C2 Naphthalene	49	18.61	1.02	12	12			
30			C2 Naphthalene	50	18.69	1.42	12	12			
31			C2 Naphthalene	51	19.08	0.47	12	12			
32			C2 Naphthalene	52	19.17	0.97	12	12			
33			C2 Naphthalene	52	19.45	0.35	12	12			
34	271	519	C15H32	53	20.02	0.93	15	32			
35	279	534	Acenaphthene	54	20.15	0.95	12	10			
36			C3 Naphthalene	55	20.47	0.66	13	14			
37	287	549	Dibenzofuran	57	20.85	2.00	12	8		1	
38			C3 Naphthalene	57	21.11	0.45	13	14			
39	287	549	C16H34	58	22.17	0.44	16	34			
40	294	561	Fluorene	59	22.32	1.44	13	10			
41			C3 Naphthalene	61	22.40	1.63	13	14			
42			C1 Dibenzofuran	61	23.10	0.47	13	10			
43			C1 Fluorene	61	24.73	0.29	14	12			
44	316	601	C18H38	62	26.15	0.64	18	38			
45	340	644	Phenanthrene	64	26.42	2.33	14	10			
46	340	644	Anthracene	65	26.62	0.55	14	10			
47	330	625	C19H40	65	28.00	0.47	19	40			
48			C1 Phenanthrene	65			15	12			
49			C1 Phenanthrene	66	28.56	0.28	15	12			
50			C1 Phenanthrene	66	28.69	0.43	15	12			
51			C1 Phenanthrene	66			15	12			
52			C1 Phenanthrene	67	29.04	0.35	15	12			
53	343	649	C20H42	67	29.77	0.46	20	42			
54	357	674	C21H44	68	31.61	0.63	21	44			
55	375	707	Fluoranthene	69	32.55	1.08	16	10			
56	369	695	C22H46	69	33.07	0.52	22	46			
57	393	739	Pyrene	69			16	10			
58	380	716	C23H48	70	34.63	0.55	23	48			
59	391	736	C24H50	70	36.11	0.45	24	50			
60	402	755	C25H52	71	[illegible]	[illegible]	[illegible]	[illegible]			

TABLE C-18.
GC/MS ANALYSES OF GPGP TAR OIL
(From WRI 8/26/87 Presentation)

Example of Compound Class	Relative Area Percentage	Carbon Number Range
Alkanes	5.84	11-27
Alkylbenzenes	9.66	7-11
Indans/Tetralins	11.93	9-14
Naphthalenes	20.47	10-15
Dihydrofluorenes	3.08	13-15
Fluorenes	5.44	13-15
Phenanthrenes/Anthracenes	6.15	15-18
Dihydropyrenes	1.58	16-17
Fluoranthenes	3.40	16-18
Crysenes	0.77	18
Cholanthrenes	0.66	20-21
Benzopyrenes	0.09	22
Hydroxy aromatics		
Phenols	4.67	8-10
Hydroxy Naphthalenes	3.28	10-13
Hydroxy Fluorenes	3.16	13-15
Total	80.18	

TABLE C-19.
COMPONENT ANALYSIS OF GREAT PLAINS TAR OIL BY LIQUID-LIQUID EXTRACTION AND ELUTION CHROMATOGRAPHY[1]

Fraction	Wt. % of Oil	Wt. % of Neutrals
Tar Bases (acid extract)	4.5	
Tar Acids (base extract)	19.8	
Neutrals (raffinate)	71.1	100
Loss[1]	4.6	
Neutrals Subfractions (in order of elution)		
Saturates	6	8
Olefins/Alkylaromatics	23	33
Alkylaromatics	9	13
Aromatics	3	4
Alkylaromatic Carbonyls	4	6
Aromatic Carbonyls	3	4
Pyrrolics	3	4
Alkyl Hydroxyaromatics	5	7
Hydroxyaromatics	13	18
Loss	1.5	2

1 Includes water and solids

(1)From March 2, 1987 WRI Quarterly Technical Progress Report

APPENDIX D

Statistical Compilation of GPGP Liquid By-Product Physical and Chemical Properties

Table D-1. GPGP Rectisol Naphtha Data

		3/1/85 Amoco	5/1/85 Chromaspec	7/12/85 Lab. Char.	7/19/85 Lab. Char.	10/14/85 Lab. Char.	10/21/85 Lab. Char.	10/28/85 Lab. Char.	11/4/85 Lab. Char.	[illegible] to [illegible]
Distillation (ASTM D-86)	LV %									
	IBP	132	147	132	131		100	108	[illegible]	[illegible]
	5	150	170	150		117				
	10	158	174	158	142	126	102	122	118	[illegible]
	20	166	180		161	140	141	128	14[illegible]	
	30	174	186	174	172	155	156	150	155	[illegible]
	50	186	197	186	187	178	178	[illegible]	[illegible]	[illegible]
	70	202	214	202	207	212	196	19[illegible]	19[illegible]	
	80	216	230	244	219	240	214	[illegible]	[illegible]	
	90	244	259	268	246	243	232	250	24[illegible]	[illegible]
	95	286	298	286	294	264	272	[illegible]	244	[illegible]
	99		301							
	% REC		98		97	94	98	98	98	
Specific Gravity	60/60 F	0.824	0.840	0.824	0.826	0.823	0.824	[illegible]	0.825	[illegible]
	API	39.[illegible]	36.4	39.[illegible]	37.8	4[illegible]	40.[illegible]	44.[illegible]	4[illegible]	[illegible]
Vapor Pressure	[illegible]		[illegible]	[illegible]		[illegible]	[illegible]	[illegible]	[illegible]	
Heating Value-HHV	Btu/lb	[illegible]	18470	17[illegible]	178[illegible]	17[illegible]	[illegible]	17[illegible]	[illegible]	
Water Content	wt%	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	
Elemental Analysis Wt%-Dry										
	C	[illegible]	[illegible]			[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	H	[illegible]	[illegible]			[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
(By Difference)	O	[illegible]	[illegible]			[illegible]	[illegible]	[illegible]	[illegible]	
	N	[illegible]	[illegible]			[illegible]	[illegible]	[illegible]	[illegible]	
	S	[illegible]	[illegible]			[illegible]	[illegible]	[illegible]	[illegible]	
	SUM	100.[illegible]	100.[illegible]			[illegible]	[illegible]	[illegible]	[illegible]	
Atomic H/C Ratio		1.40	[illegible]			[illegible]	[illegible]	[illegible]	[illegible]	
PONA	LV%									
-Paraffins		31.5	14.[illegible]							
-Olefins			[illegible]							
-Naphthenes		13.9	[illegible]							
-Aromatics		54.[illegible]	[illegible]							
Component Analysis	wt%									
Methanol			[illegible]		[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	
Acetone			4.[illegible]		[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
MEK			[illegible]		[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	
Benzene			50.[illegible]							

[illegible] 85 [illegible] Char.	11/12/85 to 2/20/86	9/1/86 Marketing	11/19/86 Lab Char	12/17/86 Lab Char
[illegible]	[illegible]	118	110	102
		131		
[illegible]	120	136	134	136
[illegible]		149	155	154
[illegible]	159	162	168	166
[illegible]	178	182	184	182
[illegible]		199	198	200
[illegible]		214	211	210
[illegible]	241	242	246	236
[illegible]	[illegible]	335	282	236
[illegible]	48	44.5	57	58
[illegible]	[illegible]	[illegible]	.819	.822
[illegible]	4[illegible]	33.5	41.0	40.5
[illegible]	[illegible]			
[illegible]		165[illegible]	[illegible]	[illegible]
[illegible]	[illegible]		[illegible]	[illegible]
[illegible]	[illegible]		55.4	54.4
[illegible]	[illegible]		[illegible]	[illegible]
[illegible]	[illegible]		4.[illegible]	[illegible]
[illegible]	[illegible]		[illegible]	[illegible]
[illegible]	[illegible]		1.5	[illegible]
[illegible]	[illegible]		[illegible]	[illegible]
[illegible]	[illegible]		1.25	1.74
		[illegible]		
		[illegible]		
		5.[illegible]		
		6.[illegible]		
[illegible]	[illegible]	[illegible]	1.5	[illegible]
[illegible]	[illegible]	4.[illegible]	.5	[illegible]
	[illegible]	[illegible]	4.6	[illegible]

Table D-1. (Continued)

		?/12/87 Lab Char	5/11/87 AMOCO	5/11/87 AMOCO (NaOH Ext)	6/18/87 AMOCO	1/20/87 UNDERC	1/22/87 UNDERC	2/2/87 UNDERC	?/?/87 Lab Char	?/?/87 API
Distillation ASTM D-86	[illegible]									
	IBP	106	111			117	104	10?		
	[illegible]		134			149	144	145		
	[illegible]	12?	142			162	156	15?		
	[illegible]	154				171	159	169		
	[illegible]	168	157			178	1?6	174		
	[illegible]	184	180			199	187	187		
	[illegible]	2??				207	20?	2??		
	[illegible]	?15				22?	22?	21?		
	[illegible]	248				270	25?	246		
	[illegible]	[illegible]						2??		
	[illegible]									
[illegible]		[illegible]								
[illegible]	[illegible]	[illegible]	[illegible]		0.?2?			[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]		4?.?			[illegible]	[illegible]	
[illegible]	[illegible]									
[illegible]	[illegible]	[illegible]							[illegible]	
[illegible]	[illegible]	[illegible]	[illegible]		[illegible]					
[illegible]										
	[illegible]	94.?	[illegible]		94.?			94.?		[illegible]
			[illegible]		[illegible]			[illegible]		
[illegible]		[illegible]	[illegible]		[illegible]			[illegible]		
		[illegible]	[illegible]		[illegible]			[illegible]		[illegible]
	[illegible]	[illegible]	[illegible]		[illegible]			[illegible]		
	[illegible]	[illegible]	[illegible]		[illegible]			[illegible]		[illegible]
[illegible]		[illegible]	[illegible]		[illegible]			[illegible]		
[illegible]	[illegible]									
[illegible]					3.?					
[illegible]					15.?					
[illegible]					[illegible]					
[illegible]					[illegible]					
Component Analysis	[illegible]									
[illegible]		[illegible]	[illegible]		[illegible]				[illegible]	
[illegible]		[illegible]	[illegible]						[illegible]	
[illegible]		4.4	[illegible]						[illegible]	
Benzene		[illegible]	45.?		46.?				41.5	
Toluene		15.?	18.?		16.1				14.?	
Xylene		[illegible]	[illegible]		0.5				[illegible]	
[illegible] Benzene										
Phenolics										

Table D-2.   GPGP Crude Phenol Data

		5/1/85 Chromaspec	10/21/85 Lab. Char.	11/4/85 Lab. Char.	11/12/85 Lab. Char.	Avg. 11/18/85 2/20/86	12/1/86 Lab. Char.	[illegible] Lab. Char.	[illegible] Lab. Char.	2/1 Lab.
Distillation (ASTM D-86)	LV %									
	IBP	211	202	196	185	200	198	200	200	
	5	258								
	10	368	370	360	370	365	348	340	365	
	20	380	375	367	375	375	368	370	375	
	30	394	380	376	382	380	375	375	384	
	50	394	400	392	394	395	386	385	394	
	70	424	430	432	424	425	417	405	466	
	80	468	475	470	470	470	464	430	520	
	90	527	520	496	476	495	508	490	600	
	95	571					520	540		
	99									
	% REC	96	91	94	92	93	96	96	91	
Specific Gravity	60/60 F	1.070	1.070	1.080	1.071	1.071	[illegible]	1.072	1.056	
	API	0.4	0.4	-0.0	0.5	[illegible]	[illegible]	0.5	[illegible]	
Vapor Pressure	RVP									
Heating Value, HHV	Btu/lb	[illegible]	14300	14400			[illegible]	[illegible]	14600	
Water Content	wt%	5.5	4.1	1.6	4.5	4.5	5.4	[illegible]	[illegible]	
Elemental Analysis wt%-Dry										
	C		82.4	78.9			77.2	79.2	[illegible]	
	H		[illegible]	[illegible]			8.2	.8	[illegible]	
(By Difference)	O		[illegible]	[illegible]			10.9	12.4	[illegible]	
	N		[illegible]	[illegible]			.4	0.4	.4	
	S		0.1	[illegible]			[illegible]	[illegible]	[illegible]	
	Sum		100.0	[illegible]			100.1	94.9	[illegible]	
Atomic H/C Ratio			[illegible]	0.98			1.01	1.18	[illegible]	
Viscosity	SUS									
	@ 80 F	51.0		500.0			70.0	62.0	260.0	
	100 F	42.0		320.0		65.0				
	212 F	36.0				50.0				
	@ 104 F cSt	8.2		112.0						
	210 F	1.5								
Component Analysis	wt%									
ATTN., Aromatics										
Phenols						32.4	26.8	32.6	29.8	
Cresols						24.4	19.5	20.6	15.8	
Methoxyphenol									1.0	
Ethylphenol							2.1	2.2	1.8	
Xylenols							4.9	4.9	4.9	
Naphthalene										

1/16/87 Lab. Char.	2/3/87 Lab. Char.	3/4/87 Lab. Char.
200	170	
365	360	
378	370	
384	377	
394	397	
406	456	
520	490	
600	520	
90	[illegible]	
1.056	1.058	1.064
3.5	[illegible]	1.5
14627	14650	14710
2.7	2.2	1.9
78.8	[illegible]	78.3
8.[illegible]	8.7	7.5
12.6	[illegible]	13.2
0.4	0.4	0.7
0.2	[illegible]	0.3
[illegible]	99.9	100.0
[illegible]	1.34	1.15
260.0	238.0	
		0.4
24.8	30.[illegible]	23.8
19.8	21.4	17.0
1.3	1.[illegible]	1.0
1.5	2.2	1.8
4.9	5.4	4.3

Table D-2. (Continued)

		4/3/87 Lab. Char.	9/25/87 Lab. Char.	2/17-5/16/87 UNDERC QUARTERLY ERCP1	ERCP2	5/11/87 Amoco
Distillation (ASTM D-86)	LV %					
	IBP		200	207	207	189
	5		205	208		217
	10		340	365	374	267
	20		365	374	381	371
	30		370	379	383	376
	50		385	385	394	385
	70		410	410	433	419
	80		450	444	468	463
	90		520	505	496	553
	95		525	509		
	99					
	% REC		94	95	94	
Specific Gravity	60/60 F	1.063	1.071	1.060	1.074	1.062
	API	1.6	0.6	2.0	0.3	1.5
Vapor Pressure	RVP					
Heating Value (HHV)	Btu/lb	13540	13630			
Water Content	wt%	5.4	4.6	4.5		5.5
Elemental Analysis wt%-Dry						
	C	75.7	75.7	75.6	76.0	77.1
	H	7.7	7.7	7.3	7.0	7.0
(By Difference)	O	16.2	16.1	16.8	16.0	14.8
	N	0.2	0.4	0.3	0.2	0.6
	S	0.2	0.1	0.0	0.0	0.1
	Sum	100.0	100.0	100.0	99.9	99.6
Atomic H/C Ratio		1.22	1.22	1.16	1.13	1.09
Viscosity	SUS					
-@ 80 F						
100 F						
212 F						
-@ 100 F	cSt		77.0			
210 F						
Component Analysis	Wt%					
BTX/Lt. Aromatics						
Phenols			36.6			
Cresols			25.2			
Methoxyphenol						
Ethylphenol						
Xylenols			9.0			
Naphthalene						

5/19/87 Amoco	86-73A WRI 2nd Quart.	86-73B WRI 2nd Quart.
		(from D-2887)
190	222	311
220		
322	307	365
369	376	366
375	379	367
385	391	375
416	420	390
458	471	392
527	507	457
		592
	92	
1.058	1.065	1.065
2.3	1.4	1.4
5.5	5.5	4.9
75.6	75.5	75.2
7.1	7.2	7.0
15.7	16.7	17.1
0.5	0.5	0.6
0.1	0.2	0.1
100.0	100.0	100.0
1.11	1.14	1.12
8.4		
15.6		
63.9		

Table D-2. (Continued)

		2/5/85 USS	12/18/84 Radian	1/20/86 Lab. Char.	2/20/86 Lab. Char.	UNKNOWN Northwest	10/1/86 Lab. Char.	12/1/86 Lab. Char
Phenolic Analyses	%							
Phenol		40.0	38.0	32.8	31.9	34.8	30.6	26.[illegible]
o-Cresol		7.6	7.8	6.5	6.2	6.7	5.7	5.[illegible]
m-Cresol		19.0	20.0	19.3	16.7	17.2	14.7	14.[illegible]
p-Cresol								
Guaiacol		1.4	1.3			1.2		
2-Ethylphenol		2.4 (1)	0.5		2.1 (1)	2.1	2.8	2.[illegible]
3-Ethylphenol								
4-Ethylphenol								
2,4/2,5 Xylenol		2.8	8.3 (2)		2.4	1.8	1.2	[illegible]
2,3/2,6 Xylenol		0.9			1.1 (1)	0.5	0.7	[illegible]
3,5 Xylenol		2.4 (1)			2.0	1.2	2.8	[illegible]
3,4 Xylenol		1.9						
Naphthalene		4.7	0.9					
Anisole								
Catechol			1.0					
Resorcinol								
Hydroquinone								

(1) 3,5 Xylenol and Ethyl phenol are assumed to be present in equal concentrations because the GC shows just one peak for both.

(2) All Xylenol isomers are reported as 2,4/2,5 Xylenol.

/86 Char.	12/17/86 Lab. Char.	1/16/87 Lab. Char.	2/3/87 Lab. Char.	3/4/87 Lab. Char.	9/25/87 Lab. Char.	12/17/87 HRI	2/17-5/16 1987 UNDERC
						(wt%)	(area %)
6.8	32.6	28.8	30.3	23.9	36.6	45.1	47.1
5.4	6.4	5.7	6.2	4.8	7.4	8.7	8.5
4.1	17.2	14.2	15.3	12.2	17.8	13.7	23.5
						9.2	
		1.3	1.3	1.0	2.0	2.7	1.9
[illegible] (1)	2.2 (1)	1.8 (1)	2.2 (1)	1.8 (1)	2.7	0.9	
						1.5	
						1.8	
[illegible]	2.2	2.3	2.5	2.0	2.9	9.2	9.6
[illegible] (1)	0.5 (1)	0.8 (1)	0.6 (1)	0.5 (1)	0.8		
[illegible]	2.2	1.9	2.2	1.9	2.7		
				0.1			
				0.2			
		1.0	1.3	0.1	0.1		
				0.1	0.9		
				0.1	0.1		

Table D-3. GPGP Tar Oil Data

		5/1/85 Chromaspec	7/12/85 Lab. Char.	7/24/85 Lab. Char.	10/4/85 Lab. Char.	10/14/85 Lab. Char.	10/21/85 Lab. Char.	11/4/85 Lab. Char.	11/12/85 1/20/86 Lab Avg.
Distillation (ASTM D-86)	LV %								
	IBP	210	210	180		150	184		210
	5	250			293	297			
	10	361	361	370	365	361	360	327	360
	20	396	396	410	406	396	405	363	400
	30	439	439	441	441	419	435	385	440
	50	535	535	540	527	477	500	424	520
	70	649	649	639	655	495	590	480	640
	80	689	689	652	700	680	620	525	65[illegible]
	90	728							
	95								
	99								
	% REC	98	80	80	80	88	89	87	8[illegible]
Specific Gravity	60/60 F	1.016	0.996	1.014	1.014	1.018	1.022	1.019	1.018
	API	7.8	10.5	8.0	8.0	7.5	7.0	7.4	7.5
Viscosity	SUS								
-70 F						270	360	200	
-80 F									
-100 F		79		520					170[illegible]
-120 F		53		320					37[illegible]
-150 F									[illegible]
-175 F									4[illegible]
Water Content	Wt%	1.8	2.5	1.7		2.3	2.2	2.7	2.5
Elemental Analysis	Wt%-Dry								
	C					88.0	90.[illegible]	84.7	
	H					8.4	8.5	7.7	
(By Difference)	O					2.2	0.7	6.9	
	N		0.7			0.8	0.4	0.7	.2
	S		0.5			0.5	0.7	0.4	0.4
	Sum					99.9	100.0	100.0	
Atomic H/C Ratio						1.15	1.13	1.10	
Heating Value(HHV)	Btu/lb	16400	16500	16500		16300	16600	16900	16[illegible]
Gross Component Analysis	Wt%								
BTX/Lt Aromatics		7.2							
Phenols		3.8							
Cresols		8.1							
Methoxyphenols									
Ethylphenol		1.7							
Xylenol		5.8							

11-12/85 1/20/86 Lab Avg.	5/30/86 DOD Lab	7/23/86 OSAGE TEST	7/23/86 OSAGE TEST	7/23/86 OSAGE TEST	12/1/86 Lab. Char.
210	200	190	190	200	190
360	380	360	360	370	364
400	420	390	390	415	393
440	460	430	410	445	433
520	550	510	500	520	510
640			600		560
690					580
					590
					600
80	60	60	60	60	
1.019	1.025	1.007	1.029	1.018	1.014
7.5	6.5	9.0	6.0	7.5	8.0
		830	1100	830	
					900
[illegible]					
[illegible]					59
60		65	74	65	
40					
1.5	2	2.1	1.7	2.2	2
	85.7	90.9	90.5	91.0	85.7
	[illegible]	8.4	8.6	8.5	9.6
	[illegible]	0.0	0.0	0.0	3.8
[illegible]	0.3	0.7	0.9	0.9	0.4
0.4	0.4	0.4	0.4	0.4	0.5
	100.0	100.4	100.4	100.8	100.0
	1.22	1.11	1.14	1.12	1.34
16600	16500	16500	16500	16500	16340
					1.3
					3.1
					0.8
					2.4

Table D-3. (Continued)

		12/18/86 Lab. Char.	1/15/87 Lab. Char.	2/3/87 Lab. Char.	3/4/87 Lab. Char.	4/13/87 Lab. Char.	--------- 5/17-8/16/87 UNDERC Quarterly F			
							ERC-1	ERC-2	ERC-3	EF
Distillation (ASTM D-86)	LV %									
	IBP	190	182	176	166	170	180	199	205	
	5						275	275	330	
	10	258	368	340	344	350	347	339	365	
	20	410	402	384	400	398	381	383	41[illegible]	
	30	442	430	410	428	425	405	410	44[illegible]	
	50	516	507	477	498	492	473	482	51[illegible]	
	70	620	604	548	584	578	511	545	59[illegible]	
	80	649		585	600	594	541	567	60[illegible]	
	90						565	577	61[illegible]	
	95									
	99									
	% REC	84	74	89	85	85	96	92	9[illegible]	
Specific Gravity	60/60 F	1.024	1.007	1.015	1.019	1.014	1.025	1.015		
	API	6.7	9.0	7.9	7.4	8.0	6.5	7.9		
Viscosity	SUS									
-70 F										
-80 F		1160	530	1530						
-100 F										
-120 F		62	69	98		59				
-150 F										
-175 F										
Water Content	Wt%	2.1	1.5	1.8	2	2.3				
Elemental Analysis Wt%-Dry										
	C	85.8	84.3	84.5	84.6	83.6	83.8	82.6		
	H	9.2	8.9	9.5	8.7	9.7	8.8	8.6		
(By Difference)	O	4.0	6.1	5.3	5.4	5.8	6.5			
	N	0.4	0.4	0.4	0.8	0.4	0.5	1.0		
	S	0.6	0.4	0.3	0.5	0.5	0.4			
	Sum	100.0	100.1	100.0	100.0	100.0	100.0			
Atomic H/C Ratio		1.29	1.27	1.35	1.23	1.39	1.27	1.25		
Heating Value(HHV)	Btu/lb	16470	16510	16440	16440	16500				
Gross Component Analysis	Wt%									
BTX/Lt Aromatics			1.8	7.6	3.6					
Phenols		1.6	1.3	1.9	1.6					
Cresols		4.2	3.5	4.4	3.7					
Methoxyphenols				0.5	0.4					
Ethylphenol		1.1	0.7	1.0	0.9					
Xylenol		3.1	2.5	3.2	2.7					

?PC Quarterly (Figure 1)			86-72A	86-72B	9/25/87
			WRI	WRI	Lab Char
ERC-3	ERC-4	ERC-5			
				from D-2887	
205	205	205	275	320	190
330	264	227	340	365	320
365	370	365	379	388	380
410	410	425	409	413	425
44[illegible]	450	450	443	440	450
510	523	540	544	502	520
590	605	630	587	601	588
600	635	670	607	660	
610			643	737	
				791	
				824	
87	81	78	89	99	72
			1.018	1.017	1.020
			7.5	7.6	7.2
					1400
					70
			1.5	1.5	3.4
			83.5	84.3	
			8.6	8.7	
			6.6	5.6	
			0.8	0.8	
			0.5	0.6	
			100.0	100.0	
			1.23	1.23	
					16200

Table D-3. Excluding Chromaspec 5/1/85 Analysis (Continued)

		5/1/85 Chromaspec	2/20/87 UNDERC Tar Oil-3	5/11/87 Amoco	6/18/87 Amoco
Distillation (ASTM D-1160)	LV %				
	IBP		96	142	139
	5		165	180	186
	10		178	196	199
	20		206		
	30		229	245	245
	50		309	334	315
	70		418	441	406
	80		489		
	90		595		
	95			649	623
	99				
	% REC		90	95	96
Specific Gravity	60/60 F		1.010	0.996	1.014
	API		8.6	10.6	8.0

D-10

GPGP By-Product Analysis : Statistical Summary of ASTM D-1160 Tar Oil Distillation Data [illegible] [illegible]

		sample size	Variance	Standard Deviation	t-factor for 95% conf	Range for 95% confidence interval MIN	MAX	AVERAGE (deg F)	95% Confidence Limit
Distillation (ASTM D-86)	LV %								
	IBP	3	441.6	21	4.303	[illegible]	178	126	[illegible]
	5	3	78.0	9	4.303	155	194	177	[illegible]
	10	3	86.0	9	4.303	168	214	191	[illegible]
	20	1	0.0	0	**********	206	206	206	
	30	3	56.9	8	4.303	221	258	240	[illegible]
	50	3	110.6	11	4.303	293	346	319	[illegible]
	70	3	210.9	15	4.303	386	458	422	[illegible]
	80	1	0.0	0	4.303	489	489	489	
	90	1	0.0	0	**********	595	595	595	
	95	2	169.0	13	12.706	519	753	636	[illegible]
	99	0	ERR	ERR	0	ERR	ERR	ERR	ERR
Specific Gravity	60/60 F	3	0.0	0	4.303	0.997	[illegible]	[illegible]	[illegible]

[illegible]	[illegible]	Range of Reported Data	
		MIN	MAX
[illegible]	41.5	[illegible]	14[illegible]
[illegible]	12.4	16[illegible]	[illegible]
[illegible]	[illegible]	178	[illegible]
[illegible]	[illegible]	2[illegible]	[illegible]
[illegible]	[illegible]	2[illegible]	245
[illegible]	8.[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	406	44[illegible]
	[illegible]	489	489
[illegible]	0.0	595	595
[illegible]	18.4	62[illegible]	649
[illegible]	ERR	ERR	ERR

Table D-3. D-1160 Distillation Data (Continued)

		5/1/85 Chromaspec	2/20/87 UNDERC Tar Oil-3	5/11/87 Amoco	6/18/87 Amoco
Distillation (ASTM D-1160)	LV %				
	IBP	141	96	142	139
	5	203	165	180	186
	10	330	178	196	199
	20	402	206		
	30	482	229	245	245
	50	543	309	334	315
	70	657	418	441	406
	80	742	489		
	90		595		
	95	819		649	[illegible]
	99				
% REC		94	[illegible]	95	96
Specific gravity	60/60 F	[illegible]	[illegible]	[illegible]	[illegible]
	API	7.8	8.6	6.5	[illegible]

D-11

SRGP By-Product Analysis : Statistical Summary of ASTM D-1160 Tar Oil Distillation Data R.D.Ross January

		Sample size	Variance	Standard Deviation	t-factor for 95% conf	Range for 95% confidence interval MIN	MAX	AVERAGE deg F.	95% Confidence Limit +/-
Distillation (ASTM D-86)	LV %								
	IBP	4	375.0	19	3.182	99	160	130	[illegible]
	5	4	185.0	14	3.182	162	205	184	22
	10	4	3845.0	62	3.182	128	325	227	[illegible]
	20	2	9604.0	98	12.706	-576	1184	304	[illegible]
	30	4	11053.7	105	3.182	130	468	300	167
	50	4	9455.2	97	3.182	220	530	375	155
	70	4	11440.5	107	3.182	317	657	487	170
	80	2	15002.0	122	12.706	-5.1	1752	616	[illegible]
	90	1	0.0	0	**********	595	595	595	0
	95	3	7554.7	87	4.303	481	913	697	216
	99	0	ERR	ERR	0	ERR	ERR	ERR	ERR

[illegible]	[illegible]	Range of Reported Data	
		Min	Max
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	40[illegible]
[illegible]	[illegible]	[illegible]	482
[illegible]	[illegible]	[illegible]	54[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	48[illegible]	[illegible]42
	[illegible]	[illegible]	595
[illegible]	[illegible]	[illegible]	619
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	1.01[illegible]	1.0[illegible]5
[illegible]	14.8	6.5	9.6

Table D-3. D-2887 Distillation Data (Continued)

		6/18/87 Amoco	86-72A WRI	86-72B WRI	7/1/86 DCD Lab.	9/15/87 Amoco
Distillation (ASTM D-2887)	LV %					
	IBP	232	200	198	225	225
	5	328	318	315		
	10	359	354	351	354	352
	20		396	395	399	
	30	434	423	420	435	454
	50	516	513	510	525	542
	70	609	617	611	637	656
	80		686	681	711	
	90	755	774	771	804	845
	95	814	834	831		
	99	891	930	927	975	1018
	% REC	100	100	100	99	
Specific Gravity	60/60 F	1.014	1.018	1.018	1.014	1.022
	API	8.0	7.5	7.5	8.0	7.0

[illegible] By-Product Data Analysis : Statistical Summary of ASTM D-2887 Tar Oil Distillation Data

		sample size	variance	Standard Deviation	t-factor for 95% conf	Range for 95% confidence interval (deg F)		AVERAGE (deg F)	95% Confidence Limit
Distillation (ASTM D-2887)	LV %								+/-
	IBP	5	199.6	14	2.776	234	198	216	17.5
	5	3	30.9	6	4.303	334	307	320	13.8
	10	5	7.6	3	2.776	357	351	354	3.4
	20	3	2.9	2	4.303	401	392	397	[illegible]
	30	5	143.0	12	2.776	448	419	433	14.8
	50	5	133.4	12	2.776	536	507	521	[illegible]
	70	5	323.2	18	2.776	648	604	626	[illegible]
	80	3	172.2	13	4.303	725	660	693	[illegible]
	90	5	1012.6	32	2.776	829	750	790	[illegible]
	95	3	77.6	9	4.303	848	804	826	[illegible]
	99	5	1928.6	44	2.776	1003	894	948	54.5
Specific Gravity	60/60 F	5	0.0	0	2.776	1.021	1.013	1.017	0.004
	API	5	0.2	0	2.776	8.1	7.1	7.6	0.5

[illegible] 26,1988

[illegible] Confidence Limit	Range of Reported Data MIN	MAX
[illegible]	198	232
[illegible]	315	328
[illegible]	351	359
[illegible]	395	399
[illegible]	420	454
[illegible]	519	542
[illegible]	609	656
[illegible]	681	711
[illegible]	755	845
[illegible]	914	934
[illegible]	891	1018
[illegible]	1.014	1.022
6.7	7.0	8.0

Table D-3. Excluding 6/18/87 Amoco Analysis Due To High Water Content (Continued)

		6/18/87 Amoco	86-72A WRI	86-72B WRI	7/1/86 DOD Lab.	9/15/87 Amoco
Distillation (ASTM D-2887)	LV %					
	IBP		200	198	225	225
	5		318	315		
	10		354	351	354	352
	20		396	395	399	
	30		423	420	435	454
	50		513	[illegible]	525	542
	70		617	611	637	656
	80		686	681	711	
	90		774	771	804	845
	95		834	831		
	99		930	927	975	1018
	% REC		100	100	99	
Specific Gravity	60/60 F		1.018	1.018	1.014	1.022
	API		7.5	7.5	8.0	7.0

GFGP By-Product Data Analysis : Statistical Summary of ASTM D-2887 Tar Oil Distillation Data Dat

		sample size	variance	Standard Deviation	t-factor for 95% conf	Range for 95% confidence interval (deg F)		AVERAGE (deg F)	-5% Confidence Limit
Distillation (ASTM D-2887)	LV %								(+/-)
	IBP	4	169.5	13	3.182	233	191	212	21
	5	2	2.3	2	12.706	330	303	317	13
	10	4	1.7	1	3.182	355	351	353	2
	20	3	2.9	2	4.303	401	392	397	[illegible]
	30	4	178.5	13	3.182	454	412	433	21
	50	4	158.3	13	3.182	543	502	523	20
	70	4	313.7	18	3.182	658	602	630	28
	80	3	172.2	13	4.303	725	660	693	33
	90	4	887.3	30	3.182	846	751	799	47
	95	2	2.3	2	12.706	846	819	833	13
	99	4	1388.3	37	3.182	1022	903	963	59
Specific Gravity	60/60 F	4	0.0	0	3.182	1.023	1.013	1.018	0.005
	API	4	0.1	0	3.182	8.1	6.9	7.5	0.6

January 26.1988

...nce	95% Confidence Limit	Range of Reported Data MIN	MAX
	(%)		
[illegible]	9.8	198	225
	4.0	315	318
[illegible]	0.6	351	354
[illegible]	1.1	395	399
[illegible]	4.9	420	454
[illegible]	3.8	510	542
[illegible]	4.5	611	656
[illegible]	4.7	681	711
[illegible]	5.9	771	845
[illegible]	1.6	831	834
[illegible]	6.2	927	1018
[illegible]	0.4	1.014	1.022
[illegible]	8.2	7.0	8.0

Atomic H/C Ratio				1.15	1.1[illegible]	[illegible]	
Heating Value HHV, Btu/lb	16400	16500	16500	16300	[illegible]	[illegible]	[illegible]
Gross Component Analysis Wt%							
BTX/Lt Aromatics	7.2						
Phenols	3.8						
Cresols	8.1						
Methoxyphenols							
Ethylphenol	1.7						
Xylenol	5.8						
Naphthalene Isomers	4.7						
CCR Wt%							

	[illegible]	[illegible]	1.14	[illegible]	1.34
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
					1.3
					[illegible]
					0.8
					2.4
					2.6

Atomic H/C Ratio		[illegible]	1.27	[illegible]	[illegible]	0.79	[illegible]	[illegible]
Heating Value (HHV)	Btu/lb	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]		
Gross Component Analysis								
	Wt%							
BTX(?) Aromatics			1.8	[illegible]	[illegible]			
Phenols		[illegible]	1.3	1.9	1.6			
Cresols		4.2	3.5	4.4	[illegible]			
Methoxyphenols				0.5	0.4			
Ethylphenol		1.1	0.7	1.0	0.9			
Xylenol		3.1	2.5	3.2	2.7			
Naphthalene Isomers				15.2	3.9			
CCR	Wt%	3.3	3.1	3.4	3.5	4.0		

	[illegible]	[illegible]	[illegible]	[illegible]	4.[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]	[illegible]	**********	[illegible]	[illegible]	[illegible]	
	[illegible]	[illegible]	55.9	8	4.[illegible]	[illegible]	[illegible]	24.	[illegible]
	[illegible]	[illegible]	[illegible]	[illegible]	4.[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]	15	4.[illegible]	[illegible]	458	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]	[illegible]	4.[illegible]	[illegible]	[illegible]	[illegible]	
	90	[illegible]	[illegible]	0	**********	555	555	555	
	95	[illegible]	164.[illegible]	10	[illegible]	519	[illegible]	[illegible]	[illegible]
	99	0	ERR	ERR	[illegible]	ERR	ERR	ERR	ERR
Specific Gravit.	60/60 F	3	0.0	[illegible]	4.[illegible]	0.987	[illegible]	[illegible]	[illegible]
	API	3	1.2	[illegible]	4.[illegible]	6.4	11.8	[illegible]	[illegible]

[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	441
	[illegible]	[illegible]	467
	[illegible]	[illegible]	595
[illegible]	[illegible]	[illegible]	649
[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	.996	1.014
[illegible]	[illegible]	[illegible]	10.6

2

	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	[illegible]	4	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	[illegible]	4	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	5	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	[illegible]	[illegible]	**********	[illegible]	[illegible]	[illegible]	
	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	[illegible]	[illegible]	ERR	ERR	[illegible]	ERR	[illegible]	ERR	ERR
Specific Gravity	[illegible]	[illegible]	[illegible]		[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	[illegible]	4	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]

[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]			[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
[illegible]	[illegible]	14.9	[illegible]	9.6

	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	[illegible]	5	[illegible]	31	2.776	629	[illegible]	[illegible]	[illegible]
	95	[illegible]	[illegible]	5	4.303	646	604	[illegible]	[illegible]
	99	[illegible]	1928.[illegible]	44	2.776	[illegible]	[illegible]	[illegible]	34.[illegible]
specific gravit.	60/60 F	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]	[illegible]
	API	5	0.2	0	2.776	6.1	[illegible]	[illegible]	[illegible]

[illegible]	[illegible]	[illegible]
[illegible]	681	711
[illegible]	755	845
[illegible]	814	834
[illegible]	891	1018
[illegible]	1.014	1.022
[illegible]	7.0	8.0

ℓ

	70	4	[illegible]	18	3.182	659	602	[illegible]	[illegible]
	80	[illegible]	[illegible]	13	4.303	725	666	693	[illegible]
	90	4	887.3	30	3.182	846	751	[illegible]	[illegible]
	95	[illegible]	2.3	2	12.706	846	819	[illegible]	[illegible]
	99	4	1388.3	37	3.182	[illegible]	903	[illegible]	[illegible]
Specific Gravity	60/60 F	4	0.0	0	3.182	1.020	1.013	[illegible]	[illegible]
	API	4	0.1	0	3.182	6.1	6.9	7.5	[illegible]

/

[illegible]	[illegible]	510	542
[illegible]	4.[illegible]	611	656
[illegible]	4.[illegible]	681	711
[illegible]	5.[illegible]	771	945
	[illegible]	821	834
[illegible]	6.[illegible]	927	1018
[illegible]	[illegible].4	1.014	1.022
[illegible]	[illegible]	7.0	8.0